

~~THOMAS C. HENRY~~

ENVIROMENTAL MONITORING PLAN

"The Demonstration of an Advanced Cyclone Coal Combustor,
with Internal Sulfur, Nitrogen and Ash Control, for the
conversion of a 23 MMBtu/hr Boiler to Coal"

DOE Grant No. DE-FC22-87PC79799

September 22, 1987

COAL TECH CORP.
P.O. BOX 154
MERION, PA. 19066

prepared for

U.S. DEPARTMENT OF ENERGY
PITTSBURGH ENERGY TECHNOLOGY CENTER
P.O. BOX 10940
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Environmental Monitoring Plan

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1. Project Summary and Introduction

Coal Tech Corp. will test a 30 million BTU per hour (MMBtu/hr) coal fired advanced cyclone combustor on an existing boiler designed for oil firing with an input heat capacity of about 23 MMBtu/hr. The project will be conducted at the Keeler Boiler Company plant in Williamsport, Pennsylvania. The project will be conducted in three distinct phases. Phase I consists primarily of activities involving design and the acquisition of necessary environmental approvals. During Phase II, Coal Tech will install standard equipment for handling and feeding pulverized coal, a conveyor for ash handling, and for the removal of particulate matter from the boiler flue gas. After installation of this equipment, Coal Tech will perform a 30 hour shakedown test. During Phase III, long term testing will be conducted. This long period testing will be divided into two periods of 470 and 400 hours each. The overall test program is the combination of shakedown and long term testing for a total of 900 hours. Between specific tests there will be data analysis and reporting. Upon completion of the test program, the test equipment will be removed from the test site at Keeler and a final report will be prepared describing the experience with testing the advanced cyclone combustor.

The boiler size used in the project is at the low thermal rating end of the potential market application of the combustor technology for retrofit to existing boilers. Therefore, the data to be obtained in the project will apply primarily to the performance and durability of the combustor, its auxiliary components, the fireside parts of the test boiler, and its environmental performance. This data can then be used in subsequent engineering, economic, and environmental analyses on the use of this technology in its major market applications, which are medium to large industrial boilers and utility boilers. Therefore, while the project will have an economic component, its primary focus will be technical and environmental.

As part of Phase I requirements the present Environmental Monitoring Plan (EMP) was developed from the EMP Outline submitted on March 9, 1987. The guidance used in preparing both the Outline and the Plan is the Synthetic Fuels Corporation Final Environmental Monitoring Plan Guide lines included in the Program Opportunity Notice, Appendix B - Model Cooperative Agreement, Attachment C - Federal

Assistance Reporting.

The major objective of the EMP is to provide a detailed description of Coal Tech's environmental compliance and supplemental monitoring tasks. This, in turn, would serve to provide operational and performance data aimed at ensuring that the demonstration project does not violate applicable environmental standards and is otherwise not detrimental to human health or the environment. In addition, the data base generated during this project should be directly applicable to evaluation of environmental controls used to mitigate problems related to the technology demonstrated, and also serve in alleviating environmental problems associated with the replication of this technology independent of site specific parameters.

The EMP contains six main sections, viz. Air Emission Monitoring, Waste Water Effluent Monitoring, Solid Waste Monitoring, Data Management Program, Data Quality Assurance/Quality Control, and Appendices. Each of the first three sections listed above, which address the media impacted by the project, is further subdivided to provide the requisite detailed descriptions of monitoring obligations or tasks. Compliance monitoring is that environmental and health monitoring required by Federal, State, and local regulatory agencies whereas supplemental monitoring is intended to provide environmental and health data for unregulated pollutants emitted from the demonstration project but not included in the permit -related compliance monitoring. Initially, supplemental monitoring would serve as a kind of survey or screening to identify any substances posing environmental hazards. Should any substances of this nature be found, further monitoring would be detailed.

Owing to the limited environmental impact of this project due to testing on an existing facility, small project size, and short test duration, as well as the existence of a substantial data base related to commercial/industrial coal handling and combustion practice, all monitoring tasks described below fall into the area of Source Monitoring. In addition, regulations that apply to safe working conditions will be maintained. No special monitoring of the work force is anticipated, with the exception of assuring test facility operation in accordance with standards appropriate to the site. The other requirements in the Environmental Monitoring Guidelines are not applicable to this project.

Appendix I provides details on the combustor/package boiler installation as well as a brief process summary. Appendix II is a more detailed overview of the process flow stream characteristics, including emission or discharge points, potential non-planned release points, monitoring points, and environmental controls on the unit. The Volume of Environmental Information, previously issued in response to Appendix J of the PON, is also included as Appendix III to provide additional information on the project, locality, and environmental impacts.

2. Air Emission Monitoring:

2.1 Compliance Monitoring

Compliance monitoring requirements are specified by the Pennsylvania Department of Environmental Resources (PA DER), Bureau of Air Quality Control. The required compliance monitoring for the site is found in Sections 123.11, 123.22, and 123.41 of the Department's Rules and Regulations, wherein the particulate limit is 0.4 lb/MMBtu, the SO₂ limit is 4 lb/MMBtu, and the opacity limit is 20%.

Modifications to the facility by the addition of a stack scrubber will result in compliance on particulate emissions and opacity whereas limestone injection for sulfur capture is expected to reduce SO₂ below the compliance limit. Since one of the technical objectives of this project is to establish performance characteristics of the combustor, it will be necessary to operate the combustor over a range of parametric test variables, some of which will, for brief periods, fall outside the range of acceptable environmental emissions. With the exception of these brief test periods, it is planned to operate the combustor within environmental standards.

The operating permit from the PA DER, Bureau of Air Quality Control, is included as Appendix IV.

2.1.1 Monitored Substances

The following list identifies substances to be compliance monitored. Also given, where applicable, are: sampling procedure, monitoring site location, frequency of sampling, equipment/analytical method, duration of monitoring task, and measurement sensitivity.

- o SO₂: stack gas will be continuously extracted from either the stack, at a location of 12 feet from the base, or from the fan discharge stack on the roof. After filtering and drying, the gas samples are directed to a Beckman Model 864 infra red (IR) industrial SO₂ meter. Measurement sensitivity is about 1% of full scale or ± 10 ppm out of 1000 ppm. This monitoring task will continue throughout the project.
- o Opacity: a Fire-eye Model FE-5 opacity meter is mounted in the stack at the 12 ft. location and continuously records % of light transmission reduction with an accuracy of about $\pm 5\%$ - units. This monitoring task will also continue throughout the project.
- o Smoke Number: a Bacharach smoke number measuring kit is planned to be used in assessing total stack solids both before and after the scrubber. Measurement locations are the same as for SO₂; sensitivity is around $\pm 10\%$. This measurement will be made periodically and/or on an "as needed" basis. As a

cross-check, smoke numbers and opacity should correlate.

2.2 Supplemental Monitoring

Owing to expected good performance of the combustor/scrubber combination with regard to the particulate standard only one particulate size measurement, with 10 micron size discrimination, is planned; (See Appendix-II).

This supplemental monitoring would consist of an EPA Method 5 traverse of the discharge stack with a cup-filter having a 10 micron cut-off. This would be performed by a commercial concern specializing in this testing. Sampling should be within 5% of isokinetic.

2.3 Additional Monitoring

Besides compliance and supplemental monitoring stack gases to be additionally monitored are O₂, CO, CO₂, NO_x, and unburnt hydrocarbons (HC). These gases will have the same sampling train and frequency of measurement as SO₂ (described in Section 2.1.1.). Analytical instruments employed are Beckman Models 755, 865 IR, 864 IR, 951A chemiluminant, and 400 respectively. Stack gas temperature will also be continuously measured/recorded by a thermocouple at the 12 ft. location. All monitoring is expected to continue throughout the project.

3. Waste Water Effluent Monitoring

3.1 Compliance Monitoring

Compliance requirements are specified by the Williamsport Sanitary Authority, in concurrence with the PA Department of Environmental Resources, Bureau of Water Quality Control. It is to be noted that due to the temporary nature of the present project, it is more cost effective to utilize a single pass water cooling system, than a recirculation system. The former results in a considerably higher water utilization rate than would be required in a permanent commercial installation. As a result, the potential exists for higher trace element discharges in the present project. Nevertheless, the compliance requirement for the present test effort will be met.

The necessary permit for water discharge has already been obtained from the Williamsport Sanitary Authority with the approval of the Bureau of Water Quality Control of PA DER; see Appendix V.

The following items must be monitored: Total water discharged into the sanitary system; total suspended solids in discharged water; the heavy metals, cadmium, copper, and selenium suspended in the water; the discharge temperature of the water; and the PH. The discharge limits are 0.5 lb of Cd/day, 1 lb of Cu/day, 0.1 lb of Se/day, maximum water temperature of 135°F and 5 pH 9.

The project will utilize approximately 1800 gallons per hour of city supplied water for cooling the combustor, for quenching and solidifying the molten slag and for operating the venturi scrubber.

The combined waste water stream will be at a temperature of about 130°F. Since this water directly contacts coal ash or slag solids in the stack scrubber and in the quench tank, some carryover of suspended materials is expected. These materials are entirely inorganic.

Most of the coal ash (about 80 %), as well as any limestone added for SO_x control, will be rejected from the combustor as molten slag which becomes a glassy inert material upon water quenching. Experience with slagging combustors indicates that over 90 % of the slag will be present as large agglomerates with about 10% fines which may be suspended in the quench water. In addition, the stack scrubber is expected to contribute another 10% to the solids loading. Thus, at maximum coal slurry and limestone rates, the suspended solids is estimated to be a maximum of 33 pounds per hour. When suspended in the 1800 gallons per hour of combines waste water, the suspended solids concentration is estimated at 2200 mg/l maximum. During test periods when no limestone is injected (which is a frequent occurrence) the solids loading is estimated to be about half this amount.

Based on coal trace element analyses and the estimated solids carryover as noted above, the estimated trace metal carryover for an eight hour test day would be 0.04, 0.38 and 0.002 lbs per day for selenium, copper and cadmium, respectively.

The waste water generated will be combined with the Keeler Boiler Company's sanitary waste and discharged to the Williamsport Sanitary Authority Central Treatment Plant.

The Central Treatment Plant is a secondary treatment level plant which utilizes an activated sludge process. The facility is rated for a maximum flow of 10.5 million gallons per day (MGD). The daily average flow is typically about 6 to 8 MGD or about 250,000 to 333,000 gallons per hour. Influent and effluent metal monitoring data provided by the Authority show that the following concentrations are present in the influent:

Cadmium	0.006 mg/l
Copper	0.143 mg/l
Nickel	0.011 mg/l
Zinc	0.138 mg/l

Based on corresponding effluent data points, the average removals through the treatment system range from 30 % for Cadmium to 80 % for Copper.

In comparison, the trace metals discharged by the project (and when diluted by the total treatment plant flow) will result in an increase of 0.0007 mg/l for selenium, 0.0006 mg/l for copper and 0.00003 mg/l for cadmium. These increases are not expected to have any measurable effects on treatment plant performance.

Increases in temperature and suspended solids are expected to be no greater than 0.5 F and 33 mg/l, respectively, when fully diluted by the total influent flow. These increases are also not expected to result in any effects on treatment plant performance. However a surcharge of \$0.14 per pound of suspended solids exceeding 200 mg/l will be charged to Keeler Boiler.

3.1.1 Monitored Substances

The following list identifies substances to be compliance monitored. Also given are: sampling procedure, monitoring site location, frequency of sampling, equipment/anaytical method, sensitivity and duration of monitoring task where appropriate.

- ° Total volume of water discharged into the sanitary sewer system will be measured in gallons by a standard water meter located in the feed line to all water circuits.
- ° Total suspended solids in discharged water will be measured by periodic collection of samples from the slag quench tank and scrubber discharge lines (see Appendix II) as directed by the Williamsport Sanitary Authority (see Appendix V). It is assumed at present that solid samples will be collected by periodic batch sampling into clean containers. Depending on solids loading this may be a daily practice until or unless suspended solids are found to be systematically within a narrow range and/or are relatively low. Actual solids collected will be determined gravimetrically on a daily basis. This monitoring task will likely continue throughout the project.

- ° The heavy metals cadmium, copper, and selenium will be determined from samples obtained under the total suspended solids protocol outlined above. Samples will be sent to a commercial chemical testing lab and analyzed for cadmium, copper and selenium by standard tests, viz. ASTM D3683. If, as expected, trace heavy metal levels are found to be well below compliance standards and/or to be a nearly constant mass fraction of the total suspended solids for a given coal, then direct heavy metals compliance monitoring, as described above, may be discontinued with Authority approval.
- ° Water discharge temperatures from the slag quench tank and scrubber discharge lines will be measured by immersion stem type, bi-metallic dial thermometers and recorded manually about every hour. This monitoring task will continue throughout the project.
- ° Water pH measurements will be made at the slag quench tank and scrubber discharge lines by using the Fisher Alkacid Full Range PH kit. These readings will be manually taken about every hour until or unless the PH is found to be systematically within a narrow range and operating conditions are nearly invariant. In addition, spot checking of PH by the commercial lab will also be requested.

3.2 Supplemental Monitoring

In addition to the compliance monitoring for cadmium, copper, and selenium initial samples will also be supplementally monitored for other trace heavy metals as shown in Appendix VI as well as carbon, nitrogen, and sulfur. All procedures are as for compliance monitoring with commercial lab analysis.

4. Solid Waste Monitoring

4.1 Compliance Monitoring

Compliance monitoring requirements are specified by the Resource Conservation and Recovery Act, and administrated by the PA Department of Environmental Resources, Bureau of Solid Waste Management. The pertinent substances that fall under this act are the leaching potential of heavy metals and cyanide in the slag, and the cyanide and sulfide reactivity to form gas phase sulfide and cyanide compounds. The evaluation of compliance is determined by preparing a Module 1 document in which the characteristics of the solid waste product are documented,

using laboratory test results as a basis. A sample copy of a Module 1 form plus instructions are given in Appendix VII.

Since the Coal Tech Combustor is a prototype unit, no slag/ash solid waste samples are presently available for reactivity and toxicity testing. Consequently, the samples must be generated during Phase II shakedown operation. However, as shown in Appendix VIII, the Bureau of Solid Waste Management has been notified about this situation and the disposal permit application process has been initiated.

The limits related to solid waste are: hydrogen sulfide and hydrogen cyanide gas releases <500 and <250 mg/kg, respectively, during reactivity testing. Various heavy metal limits for the EP Toxicity (or leaching) Test are found in Appendix VI.

Operation of the combustor at a nominal 23 MMBtu/hr will result in the generation of about 200 to 400 lb/hr of molten slag. The slag will be quenched and thus solidified into a glassy agglomerate which consists of the coal ash and reacted and unreacted limestone. All tests and analytical procedures are performed on slag samples taken at periodic intervals from the combustor site and analyzed by commercial laboratories. It should be emphasized that all slag generated will be collected and stored on site in 20 ton (nominally) dumpsters. Prior to consignment to an appropriate landfill several representative samples will be submitted for reactivity and toxicity testing. This practice can be modified with regard to frequency and number of samples as required by the PA DER, Bureau of Solid Waste Management. Some level of monitoring will be implemented throughout the project.

In this vein, toxicity tests have been performed by Coal Tech on slag samples taken from cyclone combustor tests at the Argonne National Laboratory. This slag is believed to be more reactive than the one that will be produced at the Williamsport site because the former is air quenched while the latter is water quenched. Nevertheless, the test results showed that no significant leaching of the heavy metals from the slag into the liquid leachate occurred. This will have to be corroborated with slags from the Williamsport site. However, reactivity tests of slag from the Argonne combustor yielded unsatisfactory results and no conclusion on this matter can be drawn until the slag from the Williamsport facility is available for analysis. See Appendix II for further details.

4.1.1 Monitored Substances

The following list identifies substances in or potentially in the slag to be compliance monitored.

- ° hydrogen sulfide and hydrogen cyanide gas evolution as per characteristic of reactivity CFR [261.23 (a) (5)]; methods as per EPA-SW-846, second edition, Section No. 2.1.3; see Appendix IX.
- ° heavy metals in leachate as per EP Toxicity Test Procedures, EPA-SW-846, Section 2.1.4, Method 1310 (see Appendix X). Individual metal analysis methods as per EPA-SW-846 and/or as per Appendix VII instructions.
- ° dissolved cyanide in leachate as per EP Toxicity Test Procedures (already referenced as Appendix X) with cyanide analyzed by method 9010 (see Appendix XI) as per EPA-SW-846.

4.2 Supplemental Monitoring

As part of the additional monitoring aimed at technical evaluation, described below, slag samples will be analyzed for carbon, nitrogen, and sulfur.

4.3 Additional Monitoring

This monitoring includes specification of the slag chemical composition by standard ASTM Methods on a periodic basis. This monitoring is expected to be of technical interest only and not have any impact on environmental/health issues. Specific chemical compounds will include: silicon dioxide, aluminum oxide, calcium oxide, iron oxide, potassium and sodium oxides, tin oxide, magnesium oxide, and total sulfur.

5. Data Quality Assurance/Quality Control

In order to insure technical validity of the environmental monitoring data obtained during this project the various methods of sampling, analysis and standards/calibration have been evaluated.

Outside of gas species and opacity /smoke number measurements, which relate to air monitoring, and several of the water measurements such as flow and temperature, all of the chemical analytical data for water and solid waste characterization will be provided by commercial testing laboratories. Besides adhering to specific ASTM or EPA procedures, as noted earlier, these labs follow prescribed methods for sample preparation and blank or standards verification as illustrated in Appendix XII.

As for "in house" air quality measurements, the various analytical instruments are zeroed and calibrated with certified gases prior to and during the test. Integrity of the sampling lines is ensured by periodic back-purging and separate-system oxygen

measurements to detect any leakage. The opacity meter is zeroed on clean, natural gas firing. Since the meter also provides a smoke number equivalent of opacity, the accuracy of the manual Bacharach method can be evaluated by cross-comparison. Other flow, weight, pressure, and temperature measurements will be made by standard industrial or scientific equipment.

6. Data Management Program

Almost all air and water quality compliance and supplemental monitoring data will be manually recorded on data sheets about once every hour during actual combustor operation. Stack gas concentration measurements, including SO₂, will be recorded essentially continuously, i.e. every 5-10 minutes, and stored in an automatic data logger. Opacity and stack gas temperature are recorded continuously on charts. Two exceptions are stack particulates, which will be determined on an "as needed" basis in consultation with the PA DER, Bureau of Air Quality; and total suspended solids as well as heavy metals in the discharged water, which will be collected periodically and sent to a commercial lab for analysis at a frequency to be determined in consultation with the Williamsport Sanitary Authority.

Acquisition of monitoring data on solid waste, viz. slag, will, of necessity, be an intermittent activity since sufficient material, representative of the bulk of the operation, must be accumulated prior to submission to a commercial lab for compliance testing. Based on the characterization of slag samples to be obtained during Phase II shakedown operation, it will then be possible to formulate a monitoring schedule in concert with the PA DER, Bureau of Solid Waste Management. It should be noted, however, that all solid waste will be collected and stored at the combustor test site until it has been properly characterized for appropriate disposal.

All data sheets containing any monitoring data will be duplicated and stored in a permanent file along with data logger printouts or discs containing stack gas data. Also to be included in this permanent file are any reports or analyses from commercial labs regarding compliance testing.

It should be noted that some of the air and solid waste data, which will to a large extent depend on parametric operation of the Coal Tech Combustor, will occasionally be in non-compliance due to baseline operation, i.e. the effects of the various environmental controls can only be evaluated in reference to conventional "un-controlled" operation. These baseline, possibly non-compliance, data will be recorded.

Besides baseline operation and data with environmental controls, e.g. staging, limestone injection, . . . any data obtained during "upset" conditions, e.g. failure of a component, loss of air or water flow, will be recorded. Since "upset" conditions will almost certainly automatically secure the fuel and thereby shut down the

operation, little if any quantitative data of environmental impact is expected. However, these "upsets", to the extent they bear on safe operation and technology replication, could be useful in providing practical operational guidance and as such would be reported.

Compliance reporting will be made to the Williamsport Sanitary Authority for water and to the PA DER, Bureau of Air Quality Control and Solid Waste Management. Exact reporting protocol and frequency have not yet been determined, however, copies of all compliance reports during the prior quarter will be appended to the Quarterly Reports to DOE. These reports will also include any change of compliance status including terms of the permit or violation notices. As with the Quarterly Reports, the Annual Report will conform with the guidelines given in the Federal Register, Vol. 48, No. 199, Oct. 13, 1983, P. 46684.

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APPENDIX "I"

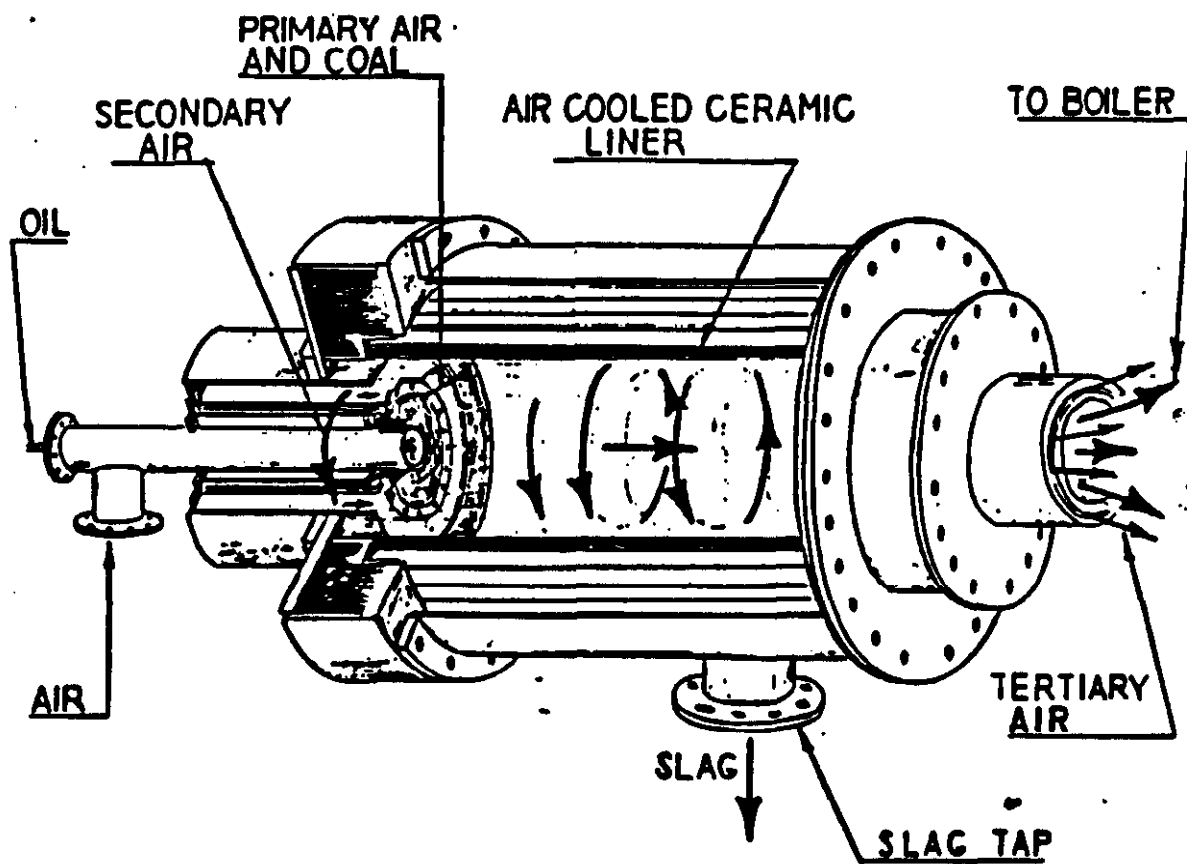
DESCRIPTION OF THE COMBUSTOR/BOILER INSTALLATION AND PROCESS SUMMARY

Figure AI.1 is a schematic diagram of the air cooled cyclone combustor. Figure AI.2 is an engineering drawing (with some details deleted) of the side and end view of the combustor installed on the 23 MMBTU/hr Keeler boiler. The figure also shows a plot plan of the combustor installation in the boiler house. Figure AI.3 is a layout of the buildings and grounds. Enclosed at the end of this Appendix is a Keeler/Dorr-Oliver commercial bulletin which describes on page four the DS-17.5 boiler being used. To-date the unit has been installed and operated for over 40 hours on coal water slurry fuel and pilot gas fuel.

Pulverized coal and combustion air are injected in the cyclone combustor at up to 21 MMBTU/hr thermal input. The combustor operates fuel rich for SO_2 and NO_x control, and final (tertiary) combustion air is injected directly into the boiler. In addition, various quantities of limestone are injected into the combustor for SO_2 control.

Coal slag and spent limestone sorbent are liquified in the combustor. Most (80-90%) of the slag mixture is drained into a water quenched tank for subsequent disposal at a landfill. It is anticipated that up to 100 hours of operation will be required before sufficient solid waste is generated for removal to the landfill. Samples from the slag will be subjected to analysis to determine compliance with solid waste disposal regulations. The balance of the slag/spent sorbent particles will be conveyed through the boiler to the stack, where they will pass through a venturi type wet scrubber which will remove sufficient particles to meet particulate emission regulations. The stack gas will also be sampled on a regular basis for compliance with air emissions regulations.

Various parts of the combustor are water cooled. This cooling water, as well as the slag quench water, and the water from the venturi scrubber will be discharged in the sanitary drains at the test site. The water discharge will be tested periodically for compliance with the thermal, suspended solids, and heavy metal trace elements standards and regulations of the Sanitary Authority.



SCHEMATIC SKETCH OF COAL TECH'S AIR COOLED CYCLONE COMBUSTOR

FIGURE AI.1

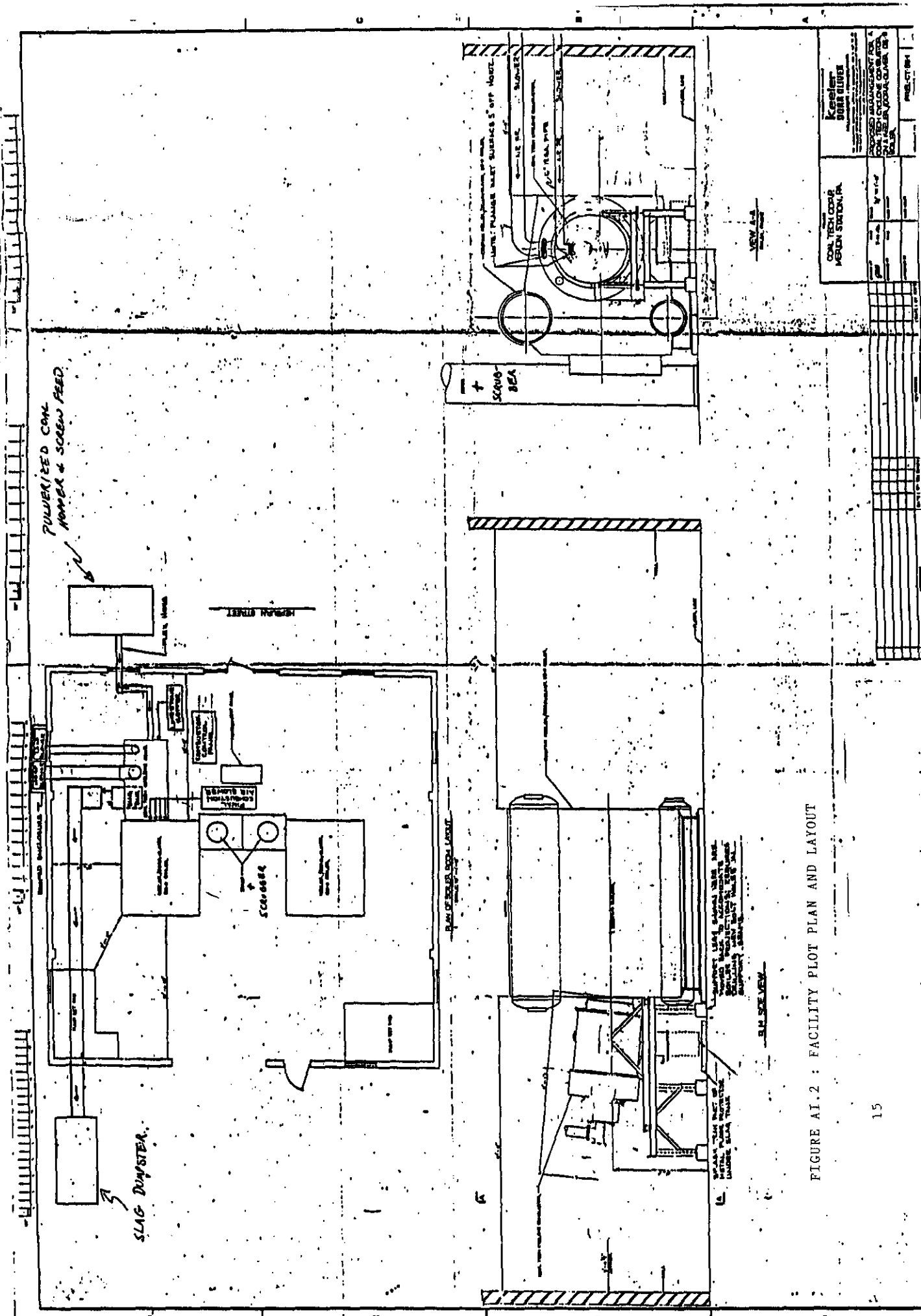


FIGURE A1.2 : FACILITY PLOT PLAN AND LAYOUT

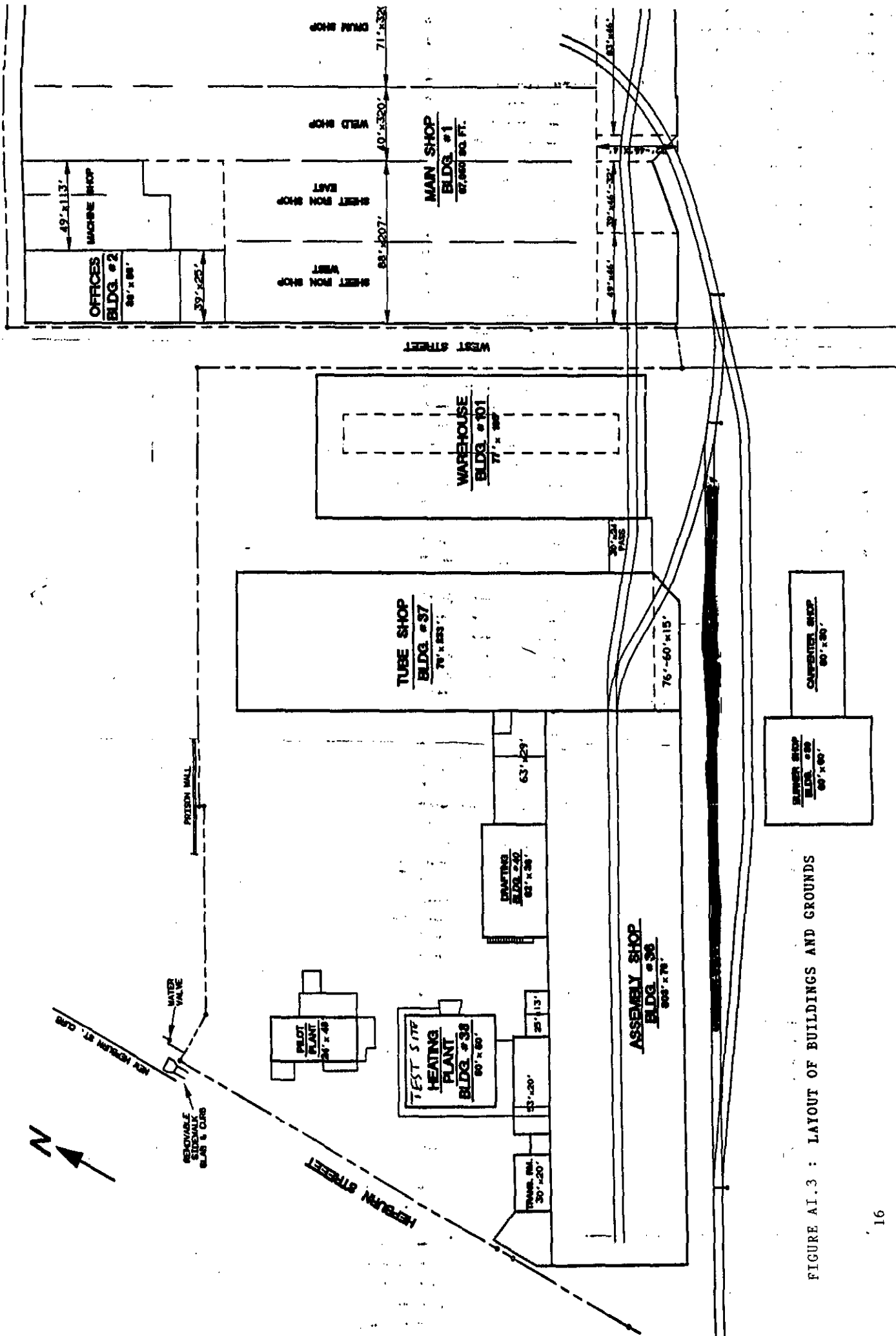
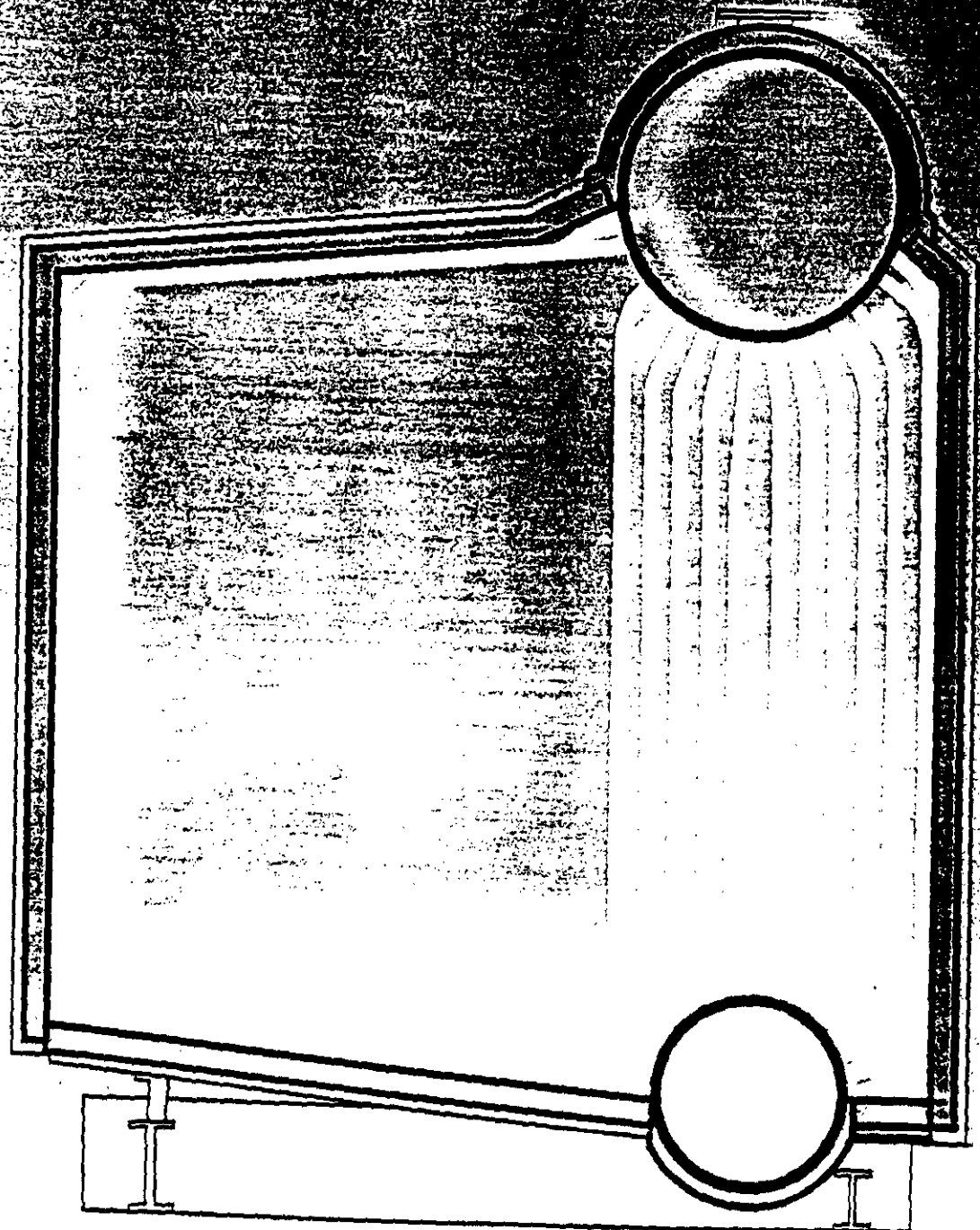


FIGURE A1.3 : LAYOUT OF BUILDINGS AND GROUNDS

Packaged Steam Generator



keeler
PACKAGED STEAM GENERATORS

**Keeler
Dorr-Oliver**



***Design for
Oil and Gas Firing***

Engineers and purchasers experienced in boiler design recognize the importance of obtaining a reliable unit that is highly efficient and requires a minimum of maintenance. To meet this need, Keeler/Dorr-Oliver, one of the leaders in the design and fabrication of industrial, commercial and institutional water tube boilers, has available a package boiler with the most advanced design double casing. This double casing design was developed to prolong casing life when firing high-sulfur-content fuel oil in a pressurized furnace.

The K/D-O "DS" unit is designed with a 10 gauge steel, seal-welded inner casing to minimize casing corrosion. The inner casing is maintained near boiler-saturated steam temperature by placing it behind and against tangent outer wall tubes, thereby maintaining the metal temperature above the dew point of the combustion gases. This eliminates condensation of the combustion gases and resultant attack due to the formation of sulphuric acid. Three inches of 1000°F. mineral wool blanket insulation is placed over this inner casing and 12 gauge steel outer casing is then installed over the insulation.

If you are considering the purchase of a package boiler and are concerned about casing corrosion, you cannot afford to overlook the many advantages that the K/D-O "DS" construction has to offer. The K/D-O "DS" casing is not

welded to the tubes in any way, resulting in ease of inspection, maintenance and repair. The design eliminates the necessity of removing the outer casing and insulation to locate or repair a gas leak at the seal weld of the inner casing. The convenient rugged double casing panels can be removed if necessary and reused. The design provides for expansion and contraction of the inner and outer casing without overheating, cracking or warping. Casings are factory-tested at 15 inches water gauge. Weather-tight design is available for outdoor installation. K/D-O does not weld tubes to each other or use widely spaced tubes with steel bars or fins welded together, providing ease of inspection, repair and maintenance. Boiler downtime will be held to a minimum with the K/D-O "DS" unit. The design is rugged and economical.

The "DS" unit is a completely packaged steam generator requiring only field service connections of fuel, water, steam and electricity. A flat concrete slab foundation and a short vent stack minimize field-installation costs.

Look over the available casing designs — compare — and you will agree that the K/D-O type "DS" design is best. We will provide the location of a K/D-O "DS" package boiler installation for you to visit and observe a unit in operation. We would like to add your name to our long list of satisfied customers.

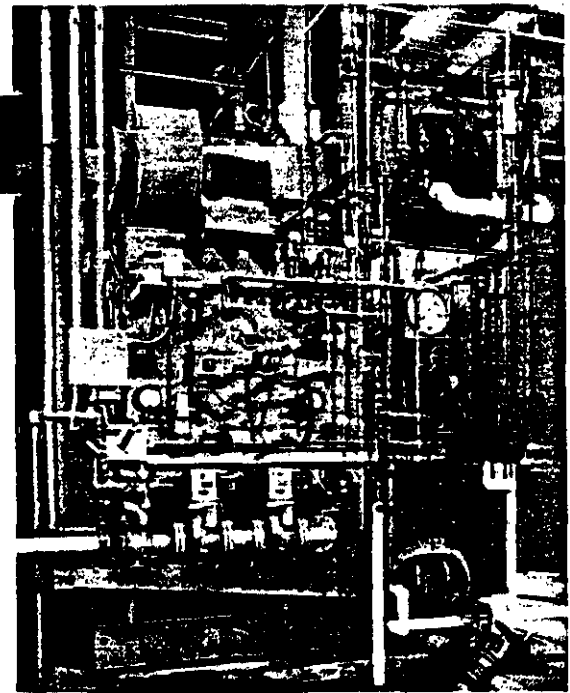
Outstanding Features of DS Casing Design Include:

- ❑ 10 gauge inner and 12 gauge outer steel casing with 3 inches of 1000°F. Mineral Wool Blanket Insulation.
- ❑ 10 gauge inner casing maintained near steam temperature by 2-inch tangent tubes to eliminate corrosion.
- ❑ Outer casing and insulation need not be disturbed to locate and repair leak at seal weld of inner casing.
- ❑ Double casing panels can be removed and reused.
- ❑ Air test can be applied to inner casing to prove tightness without disturbing outer casing or insulation.
- ❑ 15-inch water gauge shop air test proves casing tightness prior to shipment.
- ❑ Inner or outer casing is not attached to tubes in any manner.
- ❑ Simplified — most local shops can remove and install casing panels.
- ❑ Heavy inner and outer flanged casing plus channel reinforcing makes DS casing the most rugged design available.
- ❑ DS casing design provides easy access to boiler tubes.
- ❑ DS design assures longer casing life with reduced maintenance.
- ❑ Double casing installed under floor tubes.
- ❑ Heavy steel plates cover the entire front and rear of units — $\frac{3}{8}$ " front — $\frac{1}{4}$ " rear.

A Complete Modern Package

The K/D-O DS package unit is completely factory-assembled including fully integrated firing equipment, combustion and safety controls to provide reliability, and efficient operation. All you expect from a company that has specialized in building boilers for over a century.

Complete responsibility and guaranteed satisfaction are assured with the latest water tube design and a wide selection of top quality fuel burning systems to meet individual job requirements.



Additional Features

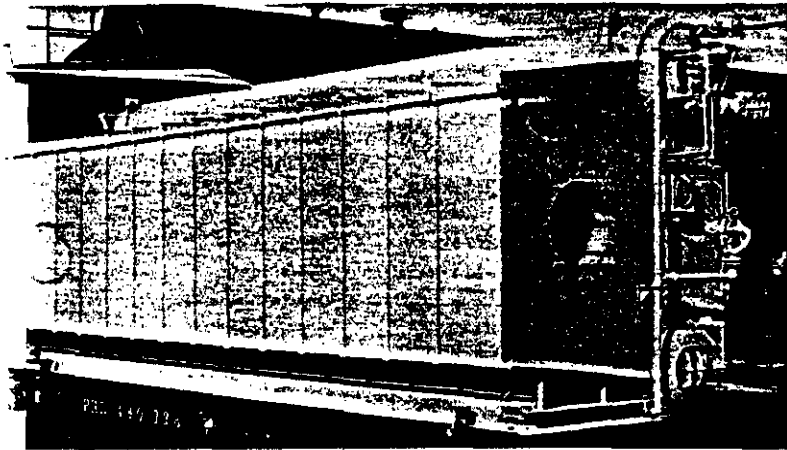
- ❑ Oil — gas or combination firing
- ❑ Standard Capacities 7500 lbs. to 100,000 lbs. per hour
- ❑ Standard Design Pressures — 200-750 psig.
- ❑ All tubes uniform diameter throughout
- ❑ Integral superheaters to 750°F.
- ❑ Economizers and air heaters available
- ❑ Complies with Military Specifications
- ❑ Available with furnace on RH or LH side
- ❑ Firing equipment and controls by Faber and other leading manufacturers
- ❑ High efficiencies
- ❑ Equipped with top quality steam and water trimmings
- ❑ Weatherproof casings available
- ❑ Steel Stub Stacks available
- ❑ Simplicity of rigging by lifting lugs from steam drum without need of special frame
- ❑ Tangent furnace tubes provide more risers than other designs resulting in improved circulation and lower tube metal temperature
- ❑ More lined feet of water cooling tubes than any other design for cooling target wall — 70% coverage with 2" bare tubes
- ❑ Drum heads extend beyond casing for inspection
- ❑ Unit is completely drainable
- ❑ Two observation doors for viewing entire furnace
- ❑ Stable water level throughout varying load conditions
- ❑ Units can be assembled at site if field conditions prohibit shipping assembled
- ❑ Standard field-erected units available — from 80,000 to 175,000 lbs. per hour with 8' and 10' wide furnaces
- ❑ Units have in-line tubes and wide and narrow space for tube removal

**Table of Standard Size Dimensions and Weights for
K/D-O "DS" Package Steam Generators**

Boiler Designation	Approx. Output lbs. Steam/Hr. (1)	HEATING SURFACE SQ. FT.			OVERALL DIMENSIONS				Furnace Width	Steam Drum Dia.	Lower Drum Dia.	Steam Conn. Dia.	Feed Conn. Dia.	Blow-off Conn. Dia.	Min. Stack Dia.	Shipping Weight lbs.
		Total (2)	ABMA (3)	Furnace Volume Cu. Ft.	A	B	C	D								
DS-8-1	7500	1541	1224	230	9'7 $\frac{3}{4}$ "	9'1 $\frac{1}{2}$ "	11'5 $\frac{1}{2}$ "	9'4 $\frac{1}{4}$ "	5'0"	36"	24"	4"	1 $\frac{1}{4}$ "	1 $\frac{1}{4}$ "	18"	29000
DS-8-2	10000	1727	1372	260	10'7 $\frac{3}{4}$ "	10'1 $\frac{3}{4}$ "	11'5 $\frac{1}{2}$ "	9'4 $\frac{1}{4}$ "	5'0"	36"	24"	4"	1 $\frac{1}{4}$ "	1 $\frac{1}{4}$ "	20"	31000
DS-8-3	12500	1944	1549	295	11'8"	11'2"	11'5 $\frac{1}{2}$ "	9'4 $\frac{1}{4}$ "	5'0"	36"	24"	4"	1 $\frac{1}{4}$ "	1 $\frac{1}{4}$ "	22"	33500
DS-15	15000	2170	1730	370	10'8 $\frac{3}{4}$ "	10'8 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	5"	1 $\frac{1}{4}$ "	1 $\frac{1}{4}$ "	24"	35500
DS-17.5	17500	2430	1945	415	11'8 $\frac{3}{4}$ "	11'2 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	5"	1 $\frac{1}{2}$ "	1 $\frac{1}{4}$ "	25"	38000
DS-20	20000	2660	2130	460	12'8 $\frac{3}{4}$ "	12'2 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	6"	1 $\frac{1}{2}$ "	1 $\frac{1}{4}$ "	27"	40500
DS-22.5	22500	2875	2300	505	13'8 $\frac{3}{4}$ "	13'2 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	6"	1 $\frac{1}{2}$ "	1 $\frac{1}{4}$ "	29"	43000
DS-25	25000	3130	2510	550	14'8 $\frac{3}{4}$ "	14'2 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	6"	1 $\frac{1}{2}$ "	1 $\frac{1}{4}$ "	30"	45500
DS-27.5	27500	3350	2690	595	15'8 $\frac{3}{4}$ "	15'2 $\frac{3}{4}$ "	12'6"	10'6"	6'0"	36"	24"	6"	1 $\frac{1}{2}$ "	1 $\frac{1}{4}$ "	31"	48500
DS-30	30000	3240	2595	625	13'8 $\frac{3}{4}$ "	13'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	6"	2"	1 $\frac{1}{2}$ "	33"	50000
DS-35	35000	3780	3040	735	15'8 $\frac{3}{4}$ "	15'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	8"	2"	1 $\frac{1}{2}$ "	36"	54000
DS-40	40000	4310	3470	845	17'8 $\frac{3}{4}$ "	17'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	8"	2"	1 $\frac{1}{2}$ "	40"	58000
DS-45	45000	4865	3925	955	19'8 $\frac{3}{4}$ "	19'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	8"	2 $\frac{1}{2}$ "	1 $\frac{1}{2}$ "	42"	65000
DS-50	50000	5365	4325	1065	21'8 $\frac{3}{4}$ "	21'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	8"	2 $\frac{1}{2}$ "	1 $\frac{1}{2}$ "	44"	69500
DS-55	55000	5925	4785	1175	23'8 $\frac{3}{4}$ "	23'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	8"	2 $\frac{1}{2}$ "	1 $\frac{1}{2}$ "	46"	74500
DS-60	60000	6485	5240	1285	25'8 $\frac{3}{4}$ "	25'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	10"	2 $\frac{1}{2}$ "	1 $\frac{1}{2}$ "	48"	80000
DS-65	65000	7025	5680	1400	27'8 $\frac{3}{4}$ "	27'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	10"	3"	2"	48"	85500
DS-70	70000	7525	6085	1525	29'8 $\frac{3}{4}$ "	29'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	10"	3"	2"	50"	90000
DS-75	75000	8085	6540	1620	31'8 $\frac{3}{4}$ "	31'2 $\frac{3}{4}$ "	13'6"	11'0"	6'6 $\frac{7}{8}$ "	36"	24"	10"	3"	2"	50"	96000
DS-10-17	50000	4580	3742	815	17'9 $\frac{1}{2}$ "	17'3 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	8"	2 $\frac{1}{2}$ "	2"	44"	56000
DS-10-18	60000	5737	4698	1030	21'9 $\frac{1}{2}$ "	21'3 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	10"	2 $\frac{1}{2}$ "	2"	47"	68000
DS-10-19	70000	6897	5657	1250	25'10 $\frac{1}{2}$ "	25'4 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	10"	3"	2"	50"	80000
DS-10-20	80000	8048	6606	1465	29'11 $\frac{1}{2}$ "	29'5 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	10"	3"	2"	53"	92000
DS-10-21	90000	8629	7087	1570	31'11 $\frac{1}{2}$ "	31'5 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	10"	3"	2"	56"	98000
DS-10-22	100000	9775	8032	1790	36'0 $\frac{1}{2}$ "	35'6 $\frac{1}{2}$ "	13'9"	12'0"	6'6"	42"	30"	10"	3"	2"	59"	110000

- 1) Capacities based on 150 psig operating pressure and 212°F. feed water temperature.
- 2) Total Circumferential Surface of all tubes
- 3) Total Circumferential Surface in Convection Surface and flat projected area in Radiant Section.

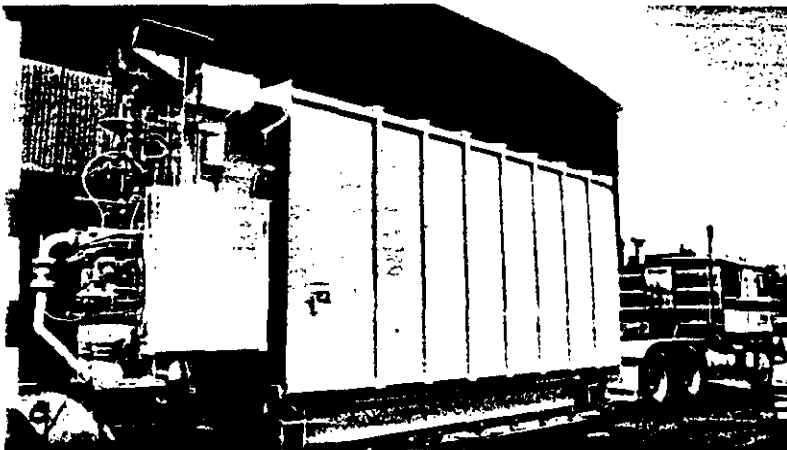
Boilers will be stamped with ABMA & ASME heating surface. Safety Valves will be selected on same basis.



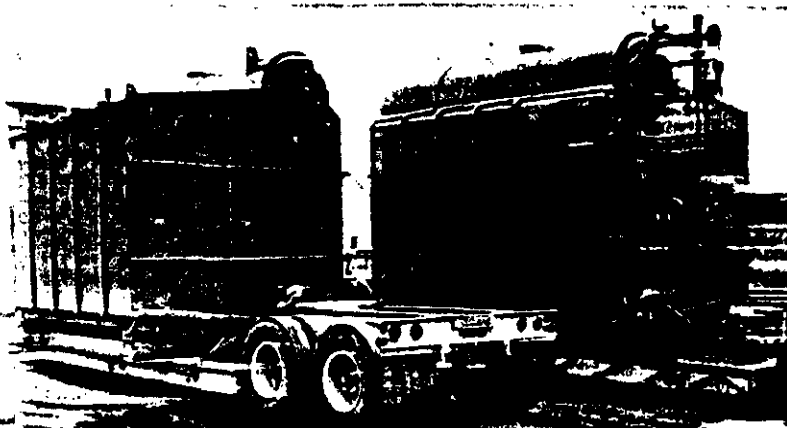
A 100,000 lb. per hr. Type DS boiler for shipment to a distiller in Scotland.



Two 80,000 lb. per hour Type DS boilers for a large tobacco processing plant.

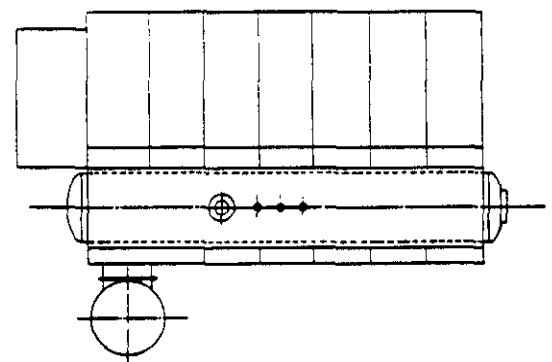


A 40,000 lb. per hour Type DS boiler ready for shipment to an automobile manufacturing facility.

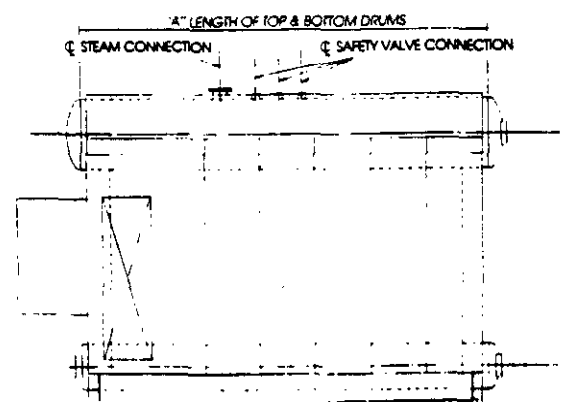
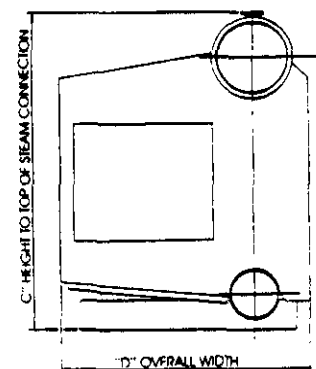


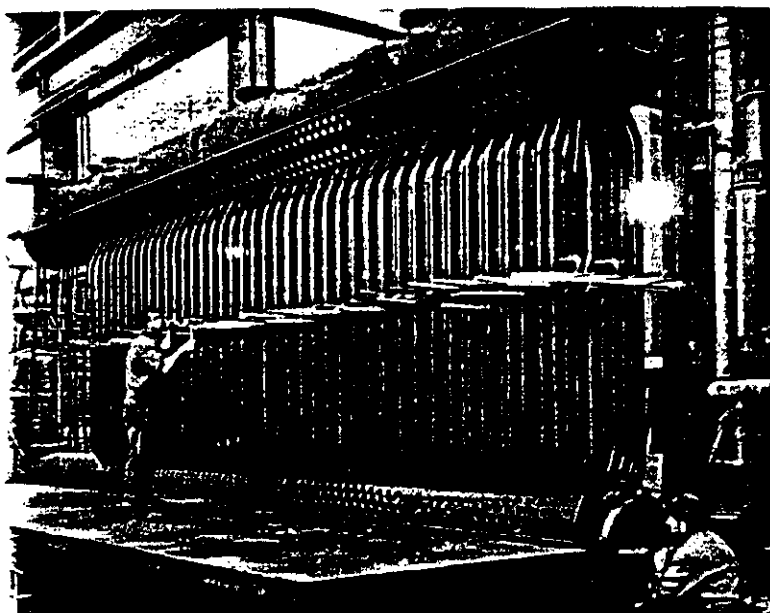
You can depend on a K/D-O Boiler for the most modern, efficient equipment for industrial, institutional or educational buildings. Whether it's a gas or oil unit, a K/D-O Boiler will give you top performance, low-cost steam and operating economy. Contact us for your nearest sales representative for complete details on a K/D-O installation.

Top View

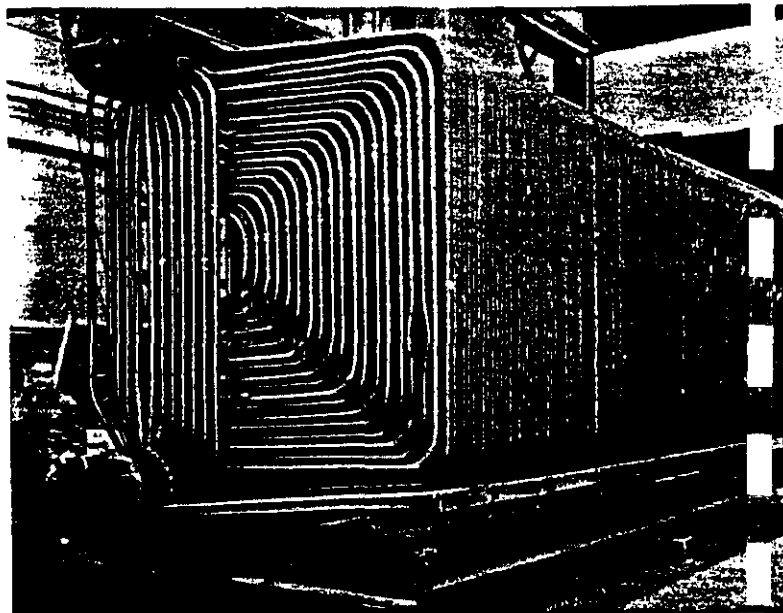


Front View

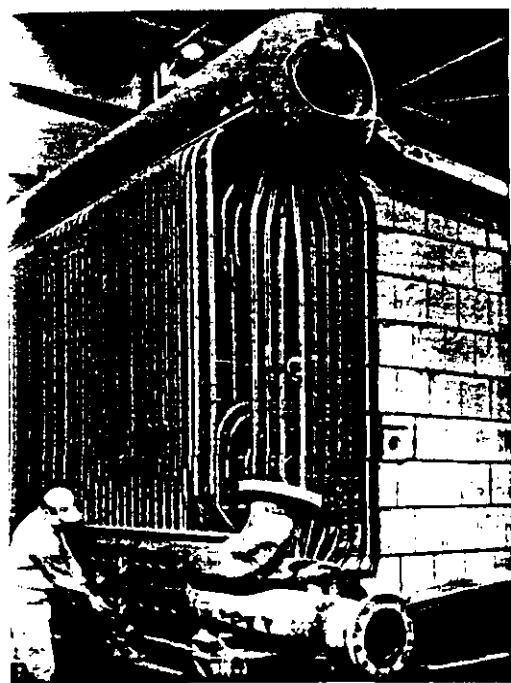




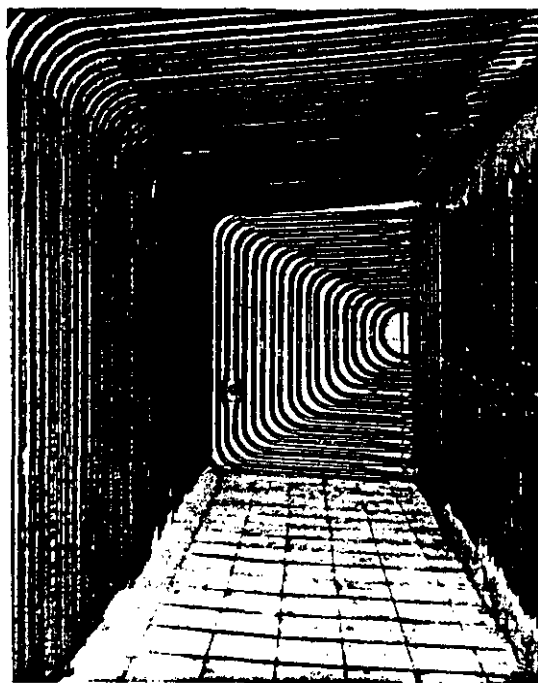
Installing tubes in Type DS package boiler.



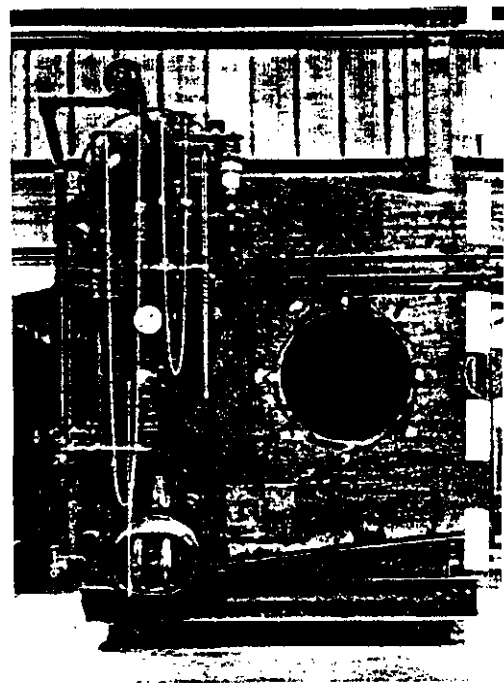
Type DS boiler being prepared for hydrostatic test.



Type DS boiler showing arrangement of superheater.



Type DS boiler showing target wall water cooling and furnace floor tile.

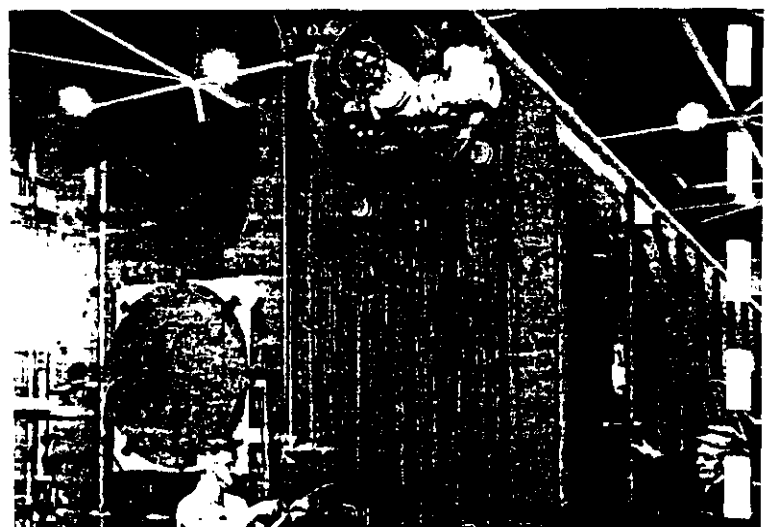


Type DS boiler, showing factory-piped steam and water trimmings.

Type DS boiler showing unique double casing.

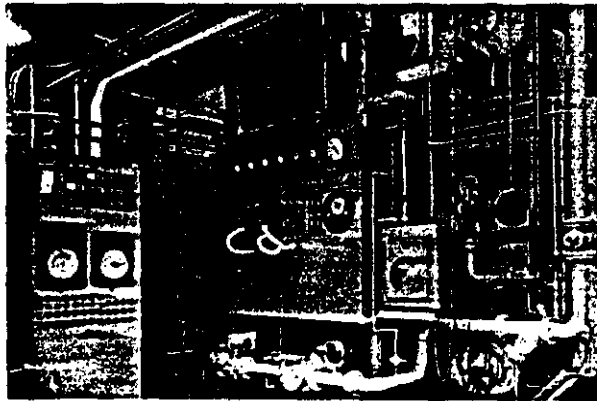


Testing casing of Type DS boiler to 15" water gauge.

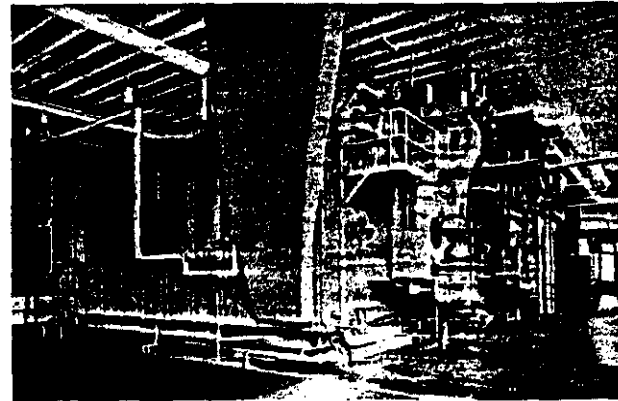




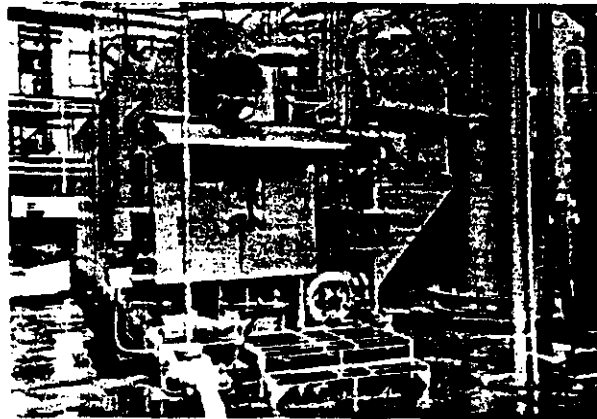
Installations



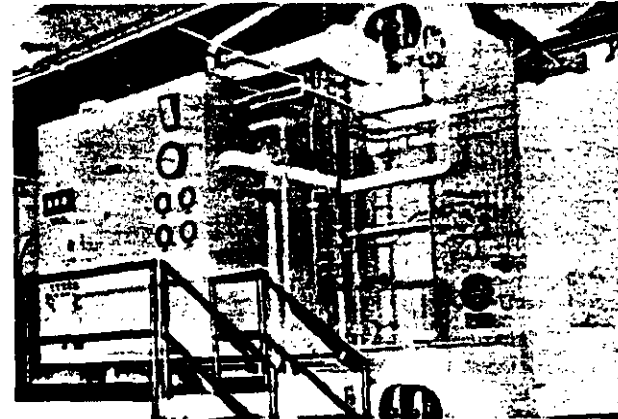
A 40,000 lb. per hour unit installed in research laboratory of building materials manufacturer.



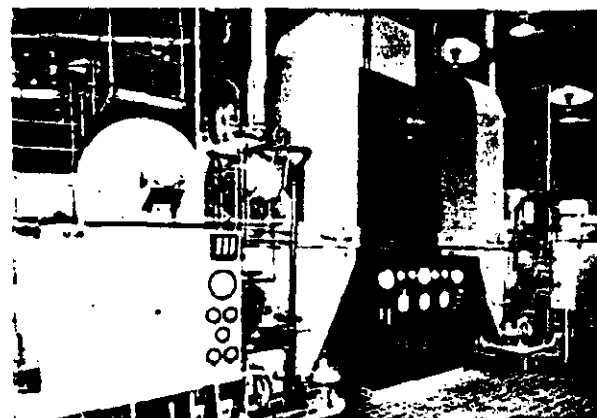
Two 44,000 lb. per hour units producing for a Southwestern university.



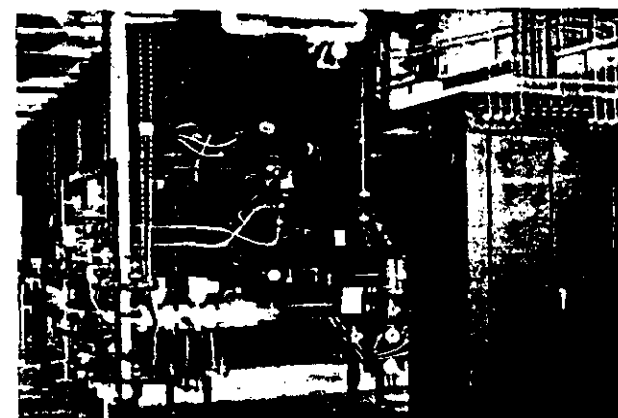
A 40,000 lb. per hour outdoor installation for a West Coast refinery.



A 100,000 lb. per hour unit installed at an atomic energy plant.



Two of three 18,000 lbs. per hour units for a Midwest hospital.



A 66,000 lb. per hour unit with superheater for space project.

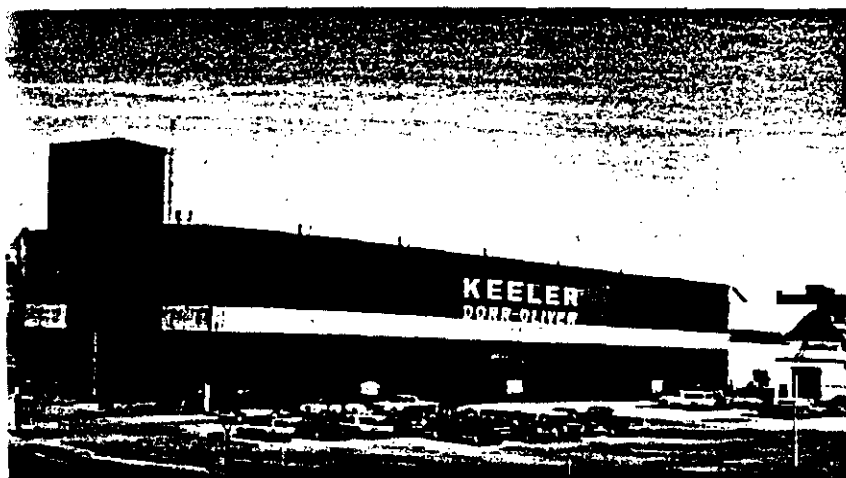
Typical Applications

- Continuous processing plants such as chemical, textile, pulp and paper, refining and breweries
- Manufacturing and assembly plants such as automotive, tire, furniture and other fabricated
- Hospitals, universities, hotels, prisons, military facilities and other institutions
- Cogenerators

Faber Burners and Control Systems

Fuel burning equipment is engineered to suit individual job requirements and all systems are completely designed, fabricated and tested by our Faber Burner Department. This provides a fully integrated boiler-burner-control unit insuring that all components of boiler-fuel burning systems are compatible, resulting in reliable single source responsibility.

- ☐ Fully packaged and shop-installed on boiler.
- ☐ Steam atomizing, air atomizing and mechanical atomizing oil burners available.
- ☐ Ring type gas burners with Stainless Steel jets to insure stable flame, optimum fuel-air ratios and maximum turndown. Gun and spud type gas burners available.
- ☐ Uni-directional air registers on smaller units to 40,000 lbs. per hour. Larger units have compound air registers.
- ☐ Single burner units up to 125,000 lbs. per hour.
- ☐ Units under 40,000 pph use single air zone; over 40,000 pph use dual. All vanes adjustable for cw or ccw rotation with single lever.
- ☐ Heavy-duty steel plate wind-box construction, $\frac{3}{8}$ " roof, reinforced. Bottom and sides $\frac{1}{4}$ ".
- ☐ Forced draft fan speed — 1750 RPM standard, insuring quiet operation and low maintenance.
- ☐ Boiler controls are front-mounted or in free-standing panel.
- ☐ 8:1 standard turndown-oil firing.
- ☐ 10:1 standard turndown-gas firing.
- ☐ 30:1 special turndown burner.
- ☐ Wide choice of combustion controls and instruments to suit individual job requirements.
- ☐ Oil-gas or combination units available for conversion of existing boiler units.
- ☐ Package type fuel oil pumping, heating and straining sets.
- ☐ Custom burner units available to burn a wide range of special fuels.
- ☐ Low NO_x burner technology proven in California.
- ☐ Standard designs meet IRI and FM requirements for safety. Others to suit are available.



Partial view of manufacturing facilities.

K/D-O designs and builds a variety of water tube boilers and is one of the oldest and most reliable water tube boiler manufacturers in America. K/D-O has faithfully served industry, institutions, utilities and government for more than century. K/D-O quality and service are known the world over. K/D-O workmanship, engineering and service are the best in the water tube boiler industry. You are invited to visit our office and plant at Williamsport to inspect our facilities and discuss your boiler requirements.

Sales Representatives In All Major Locations

Keeler

APPENDIX II

DETAILED PROCESS AND MONITORING DESCRIPTION

Figure AII.1 is a block flow diagram illustrating the liquid, solid, and gaseous effluent streams in relation to the various process flow streams and components. Also shown are the sampling point locations to be used in compliance and supplemental monitoring. The monitoring procedure to be implemented is based on Coal Tech's study of the applicable regulations as well as discussions with officials of the Williamsport Office of the PA Department of Environmental Resources.

The combustor operates as follows: Pulverized coal and combustion air are injected in the cyclone combustor at up to 21 MMBTU/hr thermal input. The combustor operates fuel rich for SO_2 and NO_x control, and final (tertiary) combustion air is injected directly into the boiler. In addition, various quantities of limestone are injected into the combustor for SO_2 control.

Coal slag and spent limestone sorbent are liquified in the combustor. Most (80-90%) of the slag mixture is drained into a water quench tank for subsequent disposal at a landfill. The balance of the slag/spent sorbent particles is conveyed through the boiler to the stack, where it will pass through a venturi type wet scrubber which will remove sufficient particles to meet particulate emission regulations. Gas sample ports in the stack, both before and after the scrubber, connect to gas analysis instruments located in a facility adjacent to the boiler house.

The main ceramic liner of the combustor is air cooled and the cooling air is used to produce swirling combustion air in the combustor. Other parts of the combustor are water cooled. This cooling water, as well as the slag quench water, and the water discharged by the venturi scrubber are all collected in a slag tank located beneath the combustor from which it is discharged in a sanitary drain at the test site. The water discharge will be tested periodically for compliance with the thermal, suspended solids, and heavy metal trace elements standards and regulations of the Sanitary Authority.

Washed coal will be delivered by PA Power & Light Co trucks to a pulverization company located within 20 miles of Williamsport. The pulverized coal will be delivered in the latter company's 20 ton pneumatic truck trailers which will re-

main at the Keeler site until empty. A blower located on the truck will convey the coal to a 5 ton storage bin. At the anticipated maximum rate of coal consumption of 0.9 tons/hr, the bin capacity is sufficient for over 5 hours of operation, a time sufficient to pick up the empty trailer, refill it at the pulverization site with 20 tons of coal and return it to Keeler before the fuel is depleted in the bin.

The bin is located adjacent to the boiler house. The bin contains a self cleaning dust collector for use during filling operations, and level indicators to control the filling. It also contains an explosion disc and an access manhole. The coal drops through a rotary valve to a smaller storage bin located above a screw feeder which meters the coal feed rate to the combustor. The coal is entrained in an eductor for delivery to the combustor. During bin refilling, attention will be given to the baghouse operation, as this is a potential non-planned release point for powdered coal due to leaks or bag perforation. In the event of such an upset, filling operations will be suspended until the problem is corrected.

The facility is currently equipped with a limestone feed system consisting of a 750 lb capacity bin, and a screw feeder with a numerically controlled feed rate adjustable from 50 to 1000 lb/hr feed rate. The limestone pneumatic feed system is being equipped with a smaller scale version of the coal feed eductor and flow splitter assembly to operate the limestone feed. In addition, a level indicator will be added to the bin. Venting of air used to refill the bin will be to the stack of the boiler, so that the limestone dust is removed by the stack scrubber.

With 2% sulfur coal, which will be used for the bulk of the test effort, the maximum limestone feed rate will be 340 lb/hr. During refilling of the bin, a manual valve located between the bin and the hopper above the screw feeder will be closed. The limestone will be emptied from 50 lb bags into a funnel which will feed into a air driven eductor. The small bin above the screw has sufficient capacity to maintain the feed rate during filling operation.

The local particulate emission standard is 0.4 lb/MMBTU. It can be met by the combined particle retention of the cyclone combustor and a venturi scrubber.

It was found in a published source that the particle exhaust from a recently operated cyclone combustor was measured to be 21% by weight below 0.5 microns and an additional 14% by weight between 0.5 and 1 microns. The particle retention in the cyclone combustor as reported for the same combustor was as high as 94%. Other data obtained verbally from DOE indicated that the stack particles from a cyclone combustor had a log normal distribution with only 10% by weight below 1 micron. Also a review of the particle retentions in cyclone combustors used in the MHD program suggested particle retentions in the 80% to the low 90% range. Cyclone combustors generally have a lower limit of 10 microns in particle retention capability. Since the particle retention efficiency of a scrubber for coarse particles is over 99% for particles above several microns, even with only several inches of H₂O pressure drop in the scrubber venturi throat, the exact particle collection efficiency of the cyclone combustor is not critical and a nominal value of 85% has been used in the scrubber selection.

The scrubber manufacturer has a proprietary computer code for analyzing the particle retention of its scrubber. The calculations indicated that scrubber pressure drops in the range of 5 to 20 inches of H₂O would meet the 0.4 lb/MMBTU emission standard. Our analysis showed that the lower pressure drop was based on the coarser particle size distribution (i.e. 10% < 1 micron). Using the finer size distribution results in a pressure drop of 15 to 20 " H₂O with the variation depending on the specific scrubber design. The unit that we have purchased requires a 15 inch pressure drop to meet the emission standard.

The nominal slag rejection rate into the slag tank will be about 350 lb/hr for the 2% sulfur coal with limestone injection. This quantity is insufficient to warrant the use of an automated slag removal system. Instead a prefabricated steel mesh slag conveyor will be placed inside the slag tank to remove the quenched slag to a dumpster located next to the slag tank. This dumpster will be rolled to another large 17-20 ton capacity dumpster to be located in the parking lot adjacent to the boiler house. A fork lift truck will be used to empty the small dumpster. When the large dumpster is filled it will be removed by an outside contractor to an approved landfill.

It is planned to monitor the stack gas emissions for NO_x and SO_2 concentration on a continuous basis throughout the test effort. To accomplish this, the extensive gas diagnostic equipment located at the Keeler Company's Fluidized Bed Boiler Test Facility, which is located adjacent to the 23 MMBTU/hr boiler, will be used. This equipment is already connected to the stack of the 23 MMBTU/hr boiler and it was used in the recently completed short duration combustor tests with coal water slurry fuel. The availability of this equipment represents a major upgrading in diagnostic equipment available to the project. The SO_2 emission standards of 4 lb/MMBTU will be met with the 2% sulfur content coal to be used in the test effort. There is no standard for NO_x emissions. Therefore, the planned use of limestone injection for SO_2 control and staged combustion for NO_x control will be aimed at meeting the New Source Performance Standards for both SO_2 and NO_x . As shown in figure AII.1, gas samples can be extracted either before or after the scrubber. This diagnostic approach is necessary to decouple the environmental control effects of the combustor and scrubber. In addition to gaseous combustion species measurements at these locations, periodic smoke number and/or opacity readings will also be obtained.

As noted the emission standard for particulates is 0.4 lb/MMBTU. To meet this standard, the cyclone combustor will remove between 80 and 94% of the coal ash. The balance will be removed with a high pressure venturi water spray scrubber which is currently being installed on the stack. Particulate standards will be attainable by combustor/boiler slag retention, in combination with the scrubber. Therefore, only one EPA Method 5 iso-kinetic measurement of particulates discharged to the atmosphere is planned as a supplemental monitoring task. Efforts will also be made to include a small particle size determination during the particulate measurement by using a cup filter with a 10 micron cut-off. Although additional particulate measurements giving total mass rate and size, both before and after the scrubber, would be of technical interest, the high cost (about \$5000 for two samples in a one day period) of performing this task by using a commercial testing concern is prohibitive for the present project and it will not be considered.

The water wastes generated from cooling of the liquid coal slag and from particle capture in the wet scrubber will be discharged into the Williamsport Sanitary Authority's system. The following items will be monitored at periodic intervals: Cumulative water discharge, total suspended solids, trace heavy me-

tals, water temperature, water pH, and water discharge temperature. The suspended solids and heavy metal concentration will be analyzed by a commercial testing laboratory. Based on preliminary tests performed on solid slags generated in a 7 MMBTU/hr slagging cyclone combustor, which was tested for Coal Tech at the Argonne National Laboratory under a DOE Contract completed on December 31, 1986, it is anticipated that the heavy metal concentration will be below the standards set by the Authority. In addition, the planned water discharge rate of about 1800 gal/hr is sufficiently low to meet the other standards set by the Authority. Continuous measurements of the water flow rate and water discharge temperature will be made. The suspended solids and heavy metal concentrations will be sampled periodically and analyzed by a commercial testing laboratory.

The solid coal slag and the sulfur bearing solid sorbent will be collected in the water filled slag tank located beneath the combustor and stored at the test site until sufficient material is collected, (about 17 tons), after which it will be disposed at the nearest approved landfill. In order to determine the the appropriate waste disposal site, a series of tests on the specific solid waste generated by the combustor must be made.

It should be emphasized that the objective of the project is to identify and operate within an envelope of conditions conducive to good environmental performance vis-avis the impacted media. Since the project is developmental, occasional operational excursions are expected to result in some effluent samples being out of environmental specifications. However, it is anticipated that the average bulk material will be within the standards. In order to fully characterize the solid waste generated, in terms of compliance and supplemental monitoring requirements, up to 17 to 20 tons of this material will be collected during Phase II and early in Phase III. This material will be stored on site. After sufficient material has been collected to assure a representative sampling procedure, the appropriate characterization tests outlined in Appendices IX to XI will be performed. The results of these tests will be used to apply for the solid waste disposal permit to PA DER.

Specifically, the following major substances will be monitored: Hydrogen sulfide and hydrogen cyanide gas evolution using EPA specified reactivity test procedures; heavy metals leaching from the solid using EPA test procedures; and dissolved cyanide in the leachate.

The above tests will be performed by a commercial test laboratory one or more times during the short duration Phase 2 test effort, and subsequently at random intervals during the Phase 3 long duration test effort.

An analysis of the leachate for the above substances was made with the slag collected in the previously cited Argonne combustor tests. The results showed that metals do not leach out of the slag. However, the slag in those tests was air quenched while in the present combustor they are water quenched. This changes the nature of the chemical bond in the solids and the reactivity of the solid waste must be determined from the Phase 2 tests. (In the recently completed coal water slurry combustor tests, the duration of limestone injection in the combustor was too short to allow the collection of sufficient slag for analysis.) Since two different coals, as well as a range of sorbent injection levels will be used, it is anticipated that periodic solid waste reactivity tests will be performed to develop a representative data base.

It is also important to note irrespective of the solid waste analytical results, this waste can be disposed at an appropriate site. Even if the slag does not meet the conditions for disposal at a local landfill, it will still be possible to dispose of the material at a landfill approved to accept for example hazardous material. Thus solid waste disposal is not a barrier problem for the implementation of this project. Of course in the unlikely event that the solid waste will require special disposal, it could reflect unfavorably on the economics of this technology.

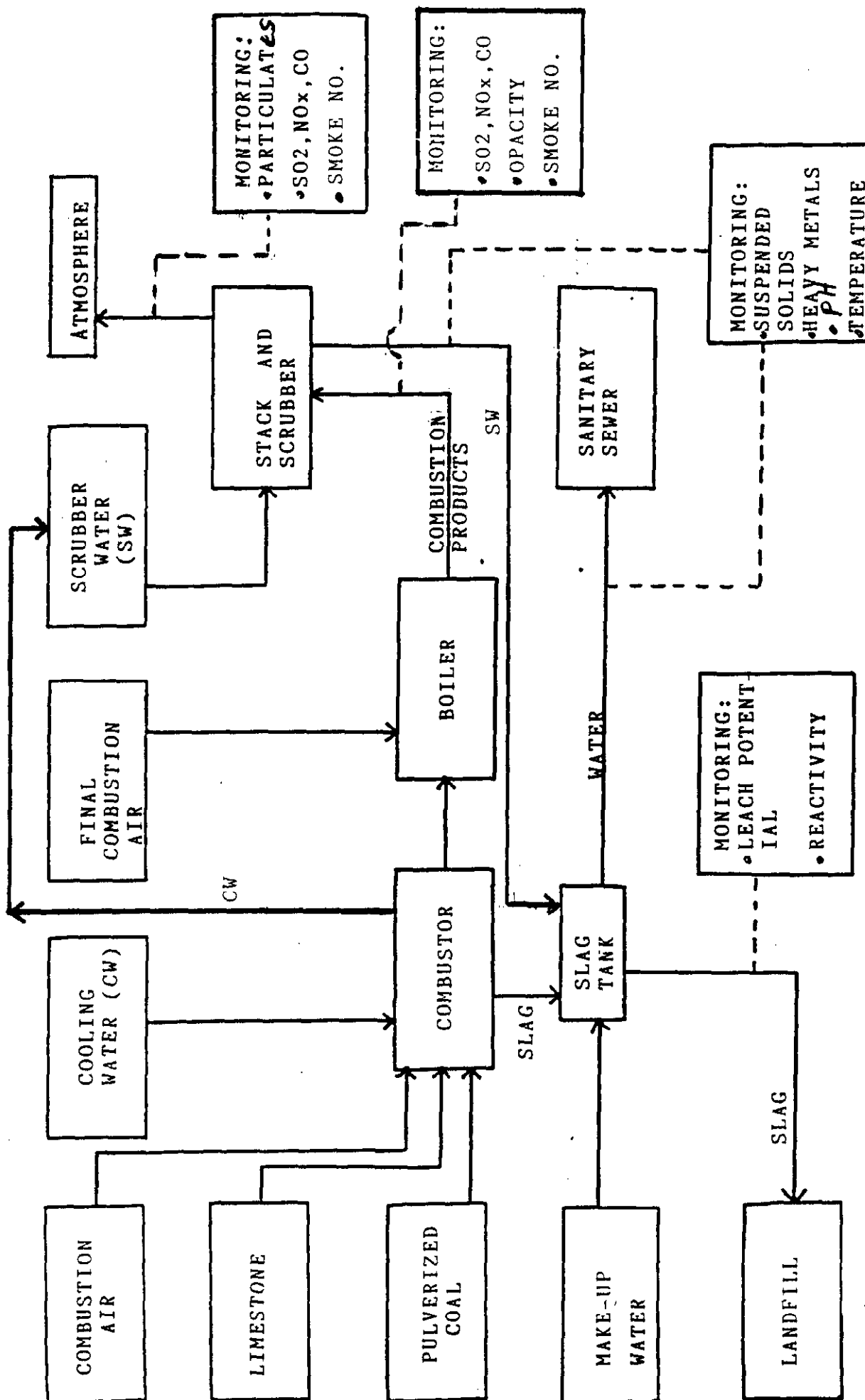


FIGURE AII.1 : PROCESS BLOCK FLOW DIAGRAM

APPENDIX III

VOLUME
OF
ENVIRONMENTAL INFORMATION

for the

Demonstration of an Advanced Cyclone Coal Combustor
with
Internal Sulfur, Nitrogen and Ash Control
for the
Conversion of a 23 MMBtu/Hour Boiler to Coal

Sponsored by:

Coal Tech, Inc.
Pennsylvania Energy Development Authority
Pennsylvania Power & Light Company

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A. DESCRIPTION OF PROPOSED ACTION

The United States Department of Energy (DOE) is proposing to enter into a cooperative agreement to conduct a cost shared Clean Coal Technology project to demonstrate the commercial feasibility of the technology for future applications. This proposed agreement is between DOE and Coal Tech, Inc. for the purpose of undertaking the project entitled "Demonstration of an Advanced Cyclone Coal Combustor with Internal Sulfur, Nitrogen, and Ash Control for the Conversion of a 23 MMBtu/Hour Boiler to Coal." Cost sharing participation with DOE will be Coal Tech, Inc., Pennsylvania Energy Development Authority, and Pennsylvania Power & Light Company.

The total estimated cost of the project is estimated to be about \$800,000, of which DOE will fund 50 percent. This endeavor is founded on the support and stimulation of the public purpose as specifically authorized by federal statute P.L. 99-190 entitled "An Act Making Appropriations for the Department of the Interior and Related Agencies for the Fiscal Year Ending September 30, 1986, and for Other Agencies."

B. DESCRIPTION OF PROPOSED PROJECT

Coal Tech, Inc. will test a 30 million BTU per hour (MMBtu/hr) coal fired advanced cyclone combustor on an existing boiler designed for oil firing with an input heat capacity of about 23 MMBtu/hr. The project will be conducted at the Keeler Boiler Company plant in Williamsport, Pennsylvania. The project will be conducted in three distinct phases. Phase I consists primarily of activities involving design and the acquisition of necessary environmental approvals. During Phase II, Coal Tech will install standard equipment for handling and feeding pulverized coal, a conveyor for slag handling, and for the removal of particulate matter from the boiler flue gas. After installation of this equipment, Coal Tech will perform a 30 hour shakedown test. During Phase III, long term testing will be conducted. This long period testing will be divided into two periods of 470 and 400 hours each. The overall test program is the combination of shakedown and long term testing for a total of 900 hours. Between specific tests there will be data analysis and reporting. Upon completion of the test program, test equipment will be removed from the test site at Keeler and a final report will be prepared describing the experience with testing the advanced cyclone combustor.

1. Purpose, Objectives and Need for the Project

This activity is being undertaken in furtherance of the DOE mission because the project involves the testing and utilization of U.S. coals that are high in sulfur and known for their slagging behavior. The activity also undertakes the long overdue testing and demonstration of a new

advanced combustor. If successful, this project will also make high sulfur U.S. coals more useful and in doing so may reduce U.S. dependence on foreign oil.

The specific objectives of the project are to:

- Demonstrate that 70 to 90 percent of the potential sulfur oxide emissions from the combustion of a 2 to 4 percent sulfur coal can be picked-up in the combustor by a sorbent.
- Demonstrate that 90 to 95 percent of the ash contained in the feed coal plus the sorbent used for SO₂ pickup can be discharged from the combustor as a low viscosity slag before it enters the boiler. (This will have the dual advantages of reducing fouling, slagging, and corrosion within the boiler and also reducing the capital requirements for particulate cleanup equipment before flue gases enter the stack for atmospheric discharge.)
- Demonstrate on a commercial scale that nitrogen oxide emissions can be reduced to 100 ppm, or less, through fuel and air staging.
- Prove that this advanced combustor has a durability in excess of 1,000 hours of on-stream, steady state operations with frequent intermittent start-ups and shutdowns.
- Develop the knowledge that this combustor is compatible with existing boilers, has a high turndown ratio, and will have the potential of bringing some existing boilers closer to meeting New Source Performance Standards economically.

2. Project Phasing and Timing

All work to be performed under the proposed agreement will be divided into three phases. Those phases, and their expected durations are:

- | | |
|-----------|-----------------------------------------------------------------------|
| Phase I | Design and Permitting (3 months) |
| Phase II | Construction and Startup (5 months) |
| Phase III | Operation, Data Collection, Reporting,
and Disposition (18 months) |

Overall program length is twenty-four months with a two-month overlap between Phases I and II. Figure 1 provides a graphic display of the time periods currently established for the project.

**U.S. DEPARTMENT OF ENERGY
FEDERAL ASSISTANCE MILESTONE PLAN**

Form Approved
GSA Gen. Reg. No. 27

1. Program/Project Identification No. DE-FC22-87PC79799		2. Program/Project Title Demonstration of an Advanced Cycles Coal Combustor	
3. Performer Name, Address Coal Tech, Inc. P.O. Box 154 Merion Station, PA 19066-0154		4. Program/Project Start Date PT 89	
5. Identification Number		5. Program/Project Completion Date	
6. Planning Category Work Breakdown Structure Total	7. Planning Category Work Breakdown Structure Detail	8. Program/Project Duration PT 87	9. Components (Name of Performer)
1.1.1.1	Environmental Information	J F M A M J J A S O N D	J A S O N D
1.1.2.1	Permit Applications & Awards		
1.1.3.1	Project Work Plan		
1.1.4.0	Design		
1.2.1.1	Program and Test Plan (See: 1.1.4.0, 1.1.3.1, 1.1.2.1)		
1.2.2	Shutdown Testing		
1.3.1.1	470 Hour Test		
1.3.1.2	470 Hour Test Analysis		
1.3.1.3	400 Hour Test		
1.3.1.4	400 Hour Test Analysis		
1.3.2	Test Facility Dismantling		
1.3.3	Project Completion		
10. Remarks		11. Signature of DOE Refining Representative and Date	

**Figure 1
Project Milestone Schedule**

3. Environmental Approvals

The environmental effects of project operation are discussed fully in Section D. As noted the project will result in the emission of air contaminants typical of fossil fuel combustion which include oxides of sulfur and nitrogen and particulates. Therefore, operation of the facility will require a State (of Pennsylvania) Plan Approval. A temporary approval has already been obtained to conduct 40 hours of parametric testing (5 days of 8 hours duration) to obtain more detailed information for the formal application. A copy of this approval is presented in Appendix A

The project will also generate waste water resulting from slag quenching and the potential need to install a small venturi scrubbing unit. Preliminary approval has been obtained from the Williamsport Sanitary Authority for discharge to the sanitary sewer system pending the results of the parametric tests. This preliminary approval is contained in Appendix A.

Solid waste in the form of boiler slag will also be generated. The offeror presently plans on disposing of this waste in the Lycoming County Sanitary Landfill. This disposal method also is dependent on analysis of the slag characteristics which will be obtained during the parametric tests.

C. SITE DESCRIPTION

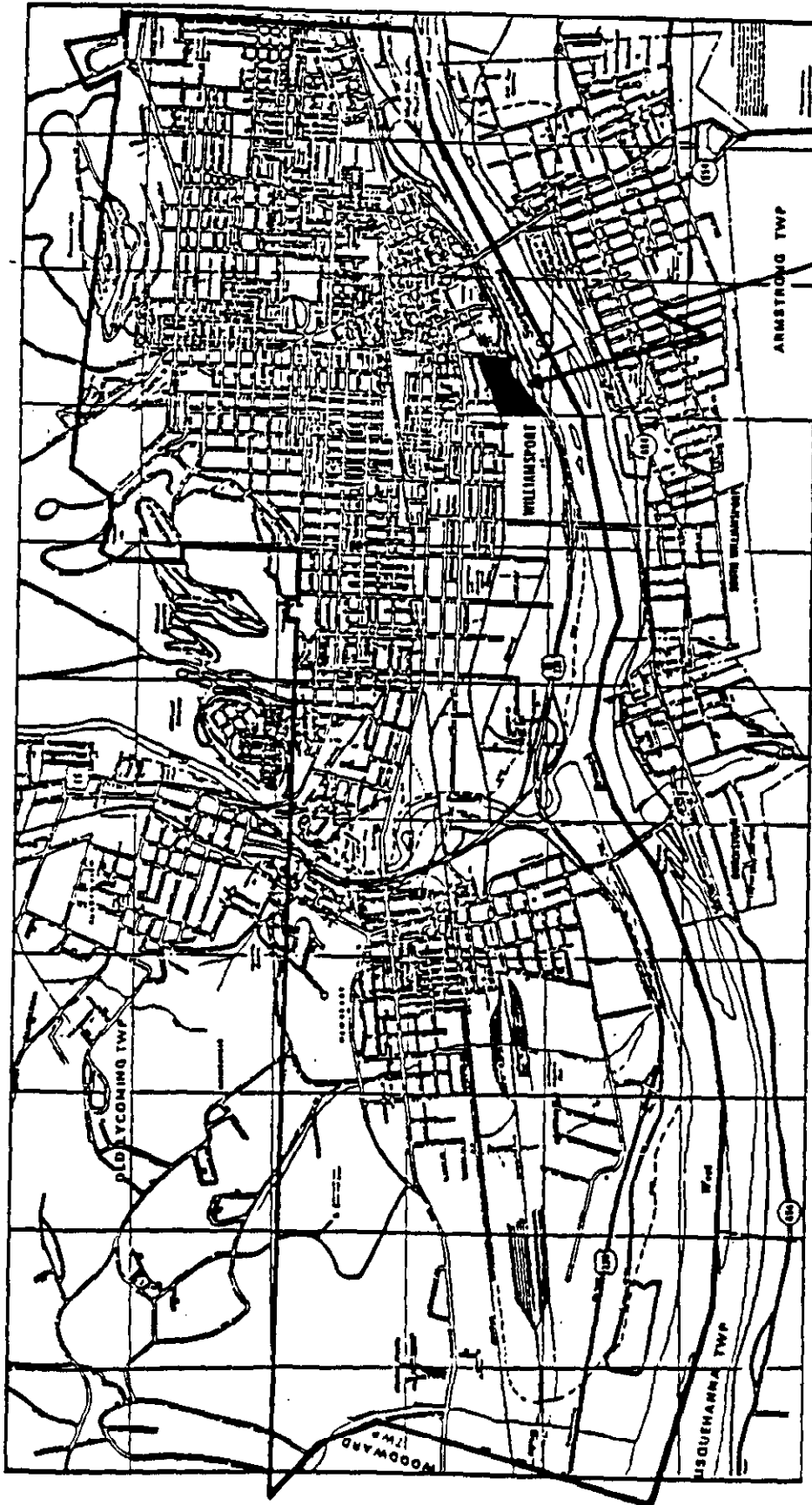
1. Geographical Location

The proposed project will be located at the Keeler Boiler Company facility in Williamsport, Pennsylvania. The City of Williamsport is situated in north-central Pennsylvania and is the largest city in Lycoming County. The population of Williamsport is approximately 33,400 residents and accounts for 28 percent of the total population of Lycoming County. The location of the proposed site is shown in Figure 2.

2. Existing Air Quality

Lycoming County is in a Non-Air Basin Area of the state as designated by the Pennsylvania Department of Environmental Resources. Lycoming County is presently classified as having attained Secondary National Ambient Air Quality Standards for all Clean Air Act Criteria Pollutants.

There are no major emission sources presently located in the County.



Keeler Boiler Co.
Property

Figure 2
City of Williamsport, Pa.

3. Existing Infrastructure and Services

The City of Williamsport is located approximately 17 miles north of Interstate 80 and can be accessed by State highways which include Routes 15 and 220. The Keeler Boiler Company property is bounded on the south by both Routes 15 and 220 and can be directly accessed without the use of city streets.

The City provides all essential services. In particular, water supply and sewage treatment are provided and available on site.

4. Existing Facilities

The Keeler Boiler Company property is fully developed and includes several buildings which house administrative personnel, fabrication operations and central heating. The proposed project will be located in the existing boiler house which contains two 23 MMBtu/hr D-tube package boilers and associated controls. These boilers are currently oil fired and are used to provide steam for space heating. One of these boilers will be converted to coal firing by the installation of the Coal Tech Advanced Combustor and associated support equipment. This facility currently has all necessary utilities in place to operate both boilers using oil as fuel.

D. POTENTIAL ENVIRONMENTAL IMPACTS

1. Construction Phase

The construction phase of the project will not involve heavy construction, land clearing or demolition. Therefore, there are no adverse impacts associated with this activity.

2. Operational Phase

- a) Air Emissions - During long term testing at full capacity, the fuel burn rate will be approximately 1 ton of coal per hour (assuming 11,000 Btu/lb of coal). Based on established emission estimates (Compilation of Air Pollutant Emission Factors, US EPA, AP-42), maximum uncontrolled emissions would be:

Particulates	80 lb/hr
SO _x (@ 2% S)	78 lb/hr
(@ 4% S)	156 lb/hr
NO _x (as NO ₂)	21 lb/hr
CO	> 1 lb/hr

The maximum annual rate will occur during the year of the 470 hour test. The total emissions for this year

(assuming uncontrolled emissions) would be:

Particulates	18.8 Tons
SOx (@ 2% S)	18.3 Tons
(@ 4% S)	36.7 Tons
NOx (as NO2)	4.9 Tons
CO	0.1 Tons

The advanced combustor, however, is expected to achieve a minimum of 90 percent particulate reduction, 70 percent SOx reduction and 80 percent NOx reduction. Therefore, the following maximum actual rates are expected:

Particulates	8.0 lbs/hr	1.9 Tons/yr	0.4 lb/MMBtu
SOx (@ 2% S)	23.4 lbs/hr	5.5 Tons/yr	1.1 lb/MMBtu
(@ 4% S)	46.8 lbs/hr	11.0 Tons/yr	2.1 lb/MMBtu
NOx (as NO2)	4.2 lbs/hr	1.0 Tons/yr	0.2 lb/MMBtu
CO	0.6 lbs/hr	0.1 Tons/yr	0.01 lb/MMBtu

In comparison to "significant net emission increases" as defined in the PSD regulations (40 CFR 52.21) which are:

Particulates	25 TPY
SO2	40 TPY
NOx	40 TPY
CO	100 TPY

the maximum controlled emissions are considerably below these significance levels.

State standards for sources located in Non-Air Basin areas limit particulate emissions to 0.4 lb/MMBtu and sulfur oxides to 4 lb/MMBtu. The expected emission rate at the minimum reduction achieved by the combustor for sulfur oxides is far below this level. However, the particulate emission rate is potentially at or above this limit. Therefore, Coal Tech proposes to install a venturi scrubber to remove fine particulates from the stack gas. The final design of the scrubber is awaiting the results of a short term parametric test to determine the actual particulate emissions which would result from actual combustor operation. The minimum removal rate will be chosen to achieve the state particulate standard with an adequate margin for safety under the maximum expected particulate loading.

- b) Liquid Wastes - The project will utilize approximately 3600 gallons per hour of city supplied water for cooling the combustor, for quenching and solidifying the molten slag and for operating the venturi scrubber.

The combined waste water stream will be at a temperature of about 100 F. Since this water directly contacts coal ash or slag solids in the stack scrubber and in the

quench tank, some carryover of suspended materials is expected. These materials are entirely inorganic.

Most of the coal ash (about 80 percent), as well as any limestone added for SOx control, will be rejected from the combustor as molten slag which becomes a glassy, inert material upon water quenching. Experience with slagging combustors indicates that over 90 percent of the slag will be present as large agglomerates with about 10 percent fines which may be suspended in the quench water. In addition, the stack scrubber is expected to contribute another 10 percent to the solids loading. Thus, at maximum coal slurry and limestone rates, the suspended solids is estimated to be a maximum of 33 pounds per hour. When suspended in the 3600 gallons per hour of combined waste water, the suspended solids concentration is estimated at 1100 mg/l maximum. During test periods when no limestone is injected (which is a frequent occurrence) the solids loading is estimated to be about half this amount.

Based on coal trace element analyses and the estimated solids carryover as noted above, the estimated trace metal carryover for an eight hour test day would be 0.04, 0.38 and 0.002 lbs per day for selenium, copper and cadmium, respectively.

As noted in Section B.3, the waste water generated will be combined with the Keeler Boiler Company's sanitary waste and discharged to the Williamsport Sanitary Authority Central Treatment Plant.

The Central Treatment Plant is a secondary treatment level plant which utilizes an activated sludge process. The facility is rated for a maximum flow of 10.5 million gallons per day (MGD). The daily average flow is typically about 6 to 8 MGD or about 250,000 to 333,000 gallons per hour. Influent and effluent metal monitoring data provided by the Authority show that the following concentrations are present in the influent:

Cadmium	0.006 mg/l
Copper	0.143 mg/l
Nickel	0.011 mg/l
Zinc	0.138 mg/l

Based on corresponding effluent data points, the average removals through the treatment system range from 30 percent for cadmium to 80 percent for copper.

In comparison, the trace metals discharged by the project (and when diluted by the total treatment plant flow) will result in an increase of 0.0007 mg/l for selenium, 0.0006 mg/l for copper and 0.00003 mg/l for

cadmium. These increases are not expected to have any measurable effects on treatment plant performance.

Increases in temperature and suspended solids are expected to be no greater than 0.5 F and 33 mg/l, respectively, when fully diluted by the total influent flow. These increases are also not expected to result in any effects on treatment plant performance. However, a surcharge of \$ 0.14 per pound of suspended solids exceeding 200 mg/l will be charged to Keeler Boiler.

- c) Solid Wastes - Operation of the combustor at a nominal 23 MMBtu/hr will result in the generation of about 200 to 400 lb/hr of molten slag. The slag will be quenched and thus solidified into a glassy agglomerate which consists of the coal ash and reacted and un-reacted limestone. Based on previous tests conducted at Argonne National Laboratory and analysis of slags resulting from steel production, the solidified slag is expected to be relatively inert with all constituents bound into the matrix. However, more detailed analysis of the slag characteristics will be performed as part of the parametric tests to determine the suitability of landfill disposal.

The quantity of slag to be disposed is relatively small in comparison to estimated domestic waste generated within the county. Based on an average per capita rate of 4 to 5 pounds per person per day, the estimated annual solid waste production of the county is 86,000 to 108,000 tons per year. In comparison, the solid waste generated over the two year test program will amount to 90 to 180 tons. Therefore, the increase in solid waste generation due to the project will be small.

The Lycoming County Sanitary Landfill, which is located in Montgomery Township, is the nearest sanitary landfill and is the primary candidate for the project's solid waste disposal. Disposal of the slag at this facility requires approval of the landfill and Pennsylvania DER. Approval will be sought during Phase I of the project.

If the Lycoming facility rejects this waste, or the tests indicate that the slag is unsuitable for sanitary landfill disposal, additional alternatives will be identified and evaluated in light of the more detailed information.

E. ENVIRONMENTAL MONITORING

All waste streams resulting from the facility's operation will be monitored during the project. The monitoring frequency

will range from continuous for SO₂, NO_x and Opacity to

intermittently for all other parameters.

The actual particulate emissions will be determined in the parametric test phase with an isokinetic probe at the stack. During the 900 hour test effort, visible emissions will be the primary regular monitoring function for particulates, with periodic stack particulate sampling to obtain an average performance baseline.

Actual trace metal behavior in the slag will be determined during the parametric test. In addition, several analyses of the slag for trace metal leaching (both long term and short term characteristics) will be performed.

As part of performance evaluation during a test, many of the parameters associated with water quality will be measured. These include exit water temperature, slag tank and scrubber water pH, as well as periodic collection and chemical analyses of suspended solids. Should any of these measurements indicate excessive conditions with regard to water quality, corrective adjustments will be made in the operation.

F. MEASURES PROPOSED TO MITIGATE ADVERSE ENVIRONMENTAL EFFECTS

All waste media will be in compliance with applicable standards. The only potential exceedance is the particulate level in the stack gas. However, Coal Tech proposes to install a venturi stack scrubber to remove fine particulates to a level below the standard of 0.4 lb/MMBtu.

Appendix A

Environmental Approvals Obtained



COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL RESOURCES

Bureau of Air Quality Control
200 Pine Street
Williamsport, PA 17701-6510

August 5, 1986

Dr. Bert Zauderer, President
Coal-Tech Corporation
P. O. Box 154
Marion Station, PA 19066-0154


Dear Dr. Zauderer:

This letter serves as Bureau of Air Quality Control approval to conduct up to five one-day tests, of up to 8 hours duration each, of your coal combustor at the Keeler/Dorr-Oliver facility in Williamsport, Pennsylvania, as described in your letter of July 29, 1986. This letter also serves as a variance from the air contaminant emission limitations specified in Sections 123.11, 123.22 and 123.41 of the Rules and Regulations of the Pennsylvania Department of Environmental Resources for the duration of these tests.

Please be advised that additional testing beyond that approved herein may necessitate a formal plan approval pursuant to Section 127.11 of the Department's Rules and Regulations and/or the installation of appropriate particulate control equipment.

Should you have any questions concerning this matter, please feel free to contact Richard L. Maxwell, Jr., Chief, Engineering Services at (717) 327-3640.

Sincerely,



Joseph W. Cooper
Regional Air Pollution Control Engineer
Williamsport Regional Office

JWC/RLM/rjh

cc: File

WILLIAMSPORT SANITARY AUTHORITY

253 WEST FOURTH STREET
WILLIAMSPORT, PENNA. 17701
(717) 323-6148

September 15, 1986

Mr. William Morton
Keeler Door-Oliver Company
238 West Street
Williamsport, PA 17701

Dear Mr. Morton:

We have reviewed the proposal by Mr. Bert Zauderer of Coal Tech Corporation as presented in his letter of September 5, 1986 concerning the discharge of quench and scrubber water from a coal combustion test at your facility. If the levels of trace elements and temperature of discharge water are as presented, we see no problems associated with the discharge to sanitary sewers.

Since the test will be conducted at the Keeler Door-Oliver plant, please confirm that all water used is measured by a meter which measures water billed to Keeler's sanitary volume account, if the water is to be discharged to sanitary sewers. Due to the discharge of concentrations of solids higher than domestic sewage, please contact our Industrial Program Coordinator, Michael Manning to arrange for sampling during the test. There will be a possible surcharge of 14¢/pound of suspended solids for levels greater than 200 mg/l during the tests billed to Keeler Door-Oliver.

If you have any questions, please contact me without hesitation.

Very truly yours,


Walter A. Nicholson
Manager, Technical Division

WAN/sl

cc: Mr. Bert Zauderer

APPENDIX IV: PERMIT FROM PA DER, BUREAU OF AIR QUALITY CONTROL
FOR TESTING OPERATION

Commonwealth of Pennsylvania
Department of Environmental Resources
Bureau of Air Quality Control

PLAN APPROVAL

Permit No.	<u>41-302-045</u>	Source &	<u>Keeler DS-9 boiler</u>
Owner	<u>Keeler/Dorr-Oliver</u>	Air	<u></u>
Operator:	<u>Coal Tech Corp.</u>	Cleaning	<u></u>
	<u></u>	Device	<u></u>
Attention:	<u>Dr. Bert Zauderer, President</u>	Location	<u>Williamsport</u>
	<u>Coal Tech Corp.</u>		<u>Lycoming County</u>

In accordance with provisions of the Air Pollution Control Act, the Act of January 8, 1960, P. L. 2119, as amended, and with Chapter 127 of the rules and regulations of the Department of Environmental Resources, the Department on JUN 02 1987 approved plans for the modification of the above indicated air contamination source.

This PLAN APPROVAL expires 5/31/89.

The plan approved is subject to the following conditions:

- (1) The boiler is to be
modified in accordance with the plans submitted with the
application (as approved herein).

See attached for additional conditions.

Notify the person noted below when the installation is completed so that the source can be inspected for issuance of an OPERATING PERMIT.

NOTE:

cc: File
Harrisburg


Regional Air Pollution Control Engineer

PLAN APPROVAL CONDITIONS
APPROVAL NO. 41-302-045
COMPANY: Keeler/Dorr-Oliver

2. This plan approval is issued for the modification of a Keeler DS-9 boiler owned by Keeler/Dorr-Oliver, said modification consisting of the installation of a cyclonic coal combustor on the boiler by Coal Tech Corp.

This plan approval is also issued for the installation of a venturi scrubber; as described in the application and supplemental materials submitted for plan approval, on said boiler.

3. The venturi scrubber shall be equipped with instrumentation to monitor water flow rate to the scrubber and pressure drop across the scrubber on a continuous basis.
4. The boiler shall be equipped with instrumentation to monitor the emission of sulfur oxides, nitrogen oxides and visible air contaminants (opacity) on a continuous basis.
5. Coal Tech Corp. shall perform such stack testing upon the modified boiler as may be requested by the Department to determine compliance with the particulate matter emission limitations of Section 123.11 and the sulfur oxides emission limitations of Section 123.22 of Chapter 123 of Article III of the Rules and Regulations of the Department of Environmental Resources.
6. The issuance of an operating permit is contingent upon the combustor and scrubber being installed, and the boiler, combustor and scrubber subsequently being maintained and operated, as specified in the application and supplemental materials submitted for plan approval and upon satisfactory demonstration that any air contaminants emitted are in compliance with the requirements specified in Sections 123.11, 123.22 and 123.41 of Chapter 123 of Article III of the Rules and Regulations of the Department of Environmental Resources, as well as in compliance with the requirements specified in, or established pursuant to, any other applicable rule or regulation contained in Article III.
7. If, at any time, it is determined that the use of the aforementioned boiler is causing the emission of particulate matter in excess of the limitations specified in Section 123.11, visible air contaminants in excess of the limitations specified in Section 123.41 or sulfur oxides in excess of the limitations specified in Section 123.22, all Sections of Chapter 123 of Article III of the Rules and Regulations of the Department of Environmental Resources, or is causing the emission of any of these or any other type of air contaminant in excess of the limitations specified in, or established pursuant to, any other applicable rule or regulation contained in Article III, the company shall take immediate steps, including the installation of additional air cleaning devices, if necessary, to reduce the emissions to within the applicable limitations.
8. The company shall immediately notify the Department of any malfunction of the source(s) or associated air cleaning device(s) which results in, or may possibly be resulting in, the emission of air contaminants in excess of the limitations specified in, or established pursuant to, any applicable rule or regulation contained in Article III of the Rules and Regulations of the Department of Environmental Resources.

PLAN APPROVAL CONDITIONS
APPROVAL NO. 41-302-045
COMPANY: Keeler/Dorr-Oliver

9. This plan approval shall also serve as a temporary operating permit in accordance with the provisions of Section 127.23 of Chapter 127 of the Rules and Regulations of the Department of Environmental Resources. This temporary operating permit is valid for a period of 120 days from completion of combustor and scrubber installation to allow for startup and debugging, provided that notification of completion of installation is given to the Department within five (5) working days of the completion date. Should a period longer than 120 days be needed a temporary operating permit renewal shall be obtained.
10. Any notification required as a result of any condition herein should be directed to: Richard L. Maxwell, Jr., Chief, Engineering Services, 200 Pine Street, Williamsport, PA 17701-6510, telephone (717) 327-3640.



COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL RESOURCES

Bureau of Air Quality Control
200 Pine Street
Williamsport, PA 17701-6510

Keeler/Dorr-Oliver
Coal Tech Corp.

Attention: Dr. Bert Zauderer, President
Coal Tech Corp.

Dear Mr. Zauderer:

Attached is a Department plan approval to construct, modify, reactivate or install an air cleaning device on an air contamination source. When the respective air contamination source construction, modification, reactivation or air cleaning device installation is completed or near completion and startup is anticipated, the Department must be notified so that a short-term temporary operating permit can be issued. During the term of this temporary operating permit, Department personnel will inspect the operation of said source(s). In addition, any testing required by the plan approval is to be conducted during this time period.

A regular Department operating permit will then subsequently be issued IF (1) the special conditions incorporated within the plan approval have all been fulfilled; (2) it has been demonstrated to the Department's satisfaction that the construction, modification, reactivation or installation was carried out as proposed in the application, and that the operation of the source(s) and any associated air pollution control equipment conforms with the operational information stated on the application, and (3) it has been demonstrated to the Department's satisfaction that the air contaminant emissions from the source(s) are in compliance with the requirements specified in, or established pursuant to, all applicable Rules and Regulations of the Department of Environmental Resources.

Please note that this plan approval contains special conditions which must be fulfilled. As failure to do so would constitute a violation of Section 127.25 of the Rules and Regulations of the Department of Environmental Resources, upon which legal enforcement action could be taken, and as such failure is also grounds for denial of issuance of a regular operating permit for the source(s), it is suggested that particular attention is paid to these special conditions.

You are advised that you have the right to appeal the issuance of this plan approval. Any appeal you may wish to make must be filed with the Environmental Hearing Board, First Floor Annex, Blackstone Building, 112 Market Street, Harrisburg, PA 17120 (717-787-3483), pursuant to the Administrative Agency Law (2 Pa. C.S., Chapters 5A and 7A) and the Act of December 3, 1970, P.L. 834 (71 P.S. §510-1 et seq.). Appeals must be filed with the Environmental Hearing Board within 30 days of service of this issuance unless the appropriate statute provides a different time period. Copies of the appeal form and the Department's regulations governing practice and procedure before the Board may be obtained from the Board.

Should you have any questions about this matter, please contact Richard L. Maxwell, Jr., Chief, Engineering Services, at (717) 327-3637

Very truly yours,



Joseph W. Cooper
Regional Air Pollution Control Engineer
Williamsport Regional Office

APPENDIX V: Permit from Williamsport Sanitary Authority
for Water Discharge into Sanitary Sewer
System.

WILLIAMSPORT SANITARY AUTHORITY

123 WEST FOURTH STREET
WILLIAMSPORT, PENNA. 17701
(717) 323-6148

September 15, 1986

Mr. William Morton
Keeler Door-Oliver Company
238 West Street
Williamsport, PA 17701

Dear Mr. Morton:

We have reviewed the proposal by Mr. Bert Zauderer of Coal Tech Corporation as presented in his letter of September 5, 1986 concerning the discharge of quench and scrubber water from a coal combustion test at your facility. If the levels of trace elements and temperature of discharge water are as presented, we see no problems associated with the discharge to sanitary sewers.

Since the test will be conducted at the Keeler Door-Oliver plant, please confirm that all water used is measured by a meter which measures water billed to Keeler's sanitary volume account, if the water is to be discharged to sanitary sewers. Due to the discharge of concentrations of solids higher than domestic sewage, please contact our Industrial Program Coordinator, Michael Manning to arrange for sampling during the test. There will be a possible surcharge of 14¢/pound of suspended solids for levels greater than 200 mg/l during the tests billed to Keeler Door-Oliver.

If you have any questions, please contact me without hesitation.

Very truly yours,


Walter A. Nicholson
Manager, Technical Division

WAN/sl

cc: Mr. Bert Zauderer

APPENDIX VI. MAXIMUM CONCENTRATION OF CONTAMINANTS
FOR CHARACTERISTIC OF EP TOXICITY(a)

EPA Hazardous Waste Number	Contaminant	Maximum concentration (mg/l)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin (1,2,3,4,10,10-Hexachloro-1 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1 4-endo, endo-5,8-dimethanonaph- thalene)	0.02
D013	Lindane (1,2,3,4,5,6- Hexachlorocyclohexane, gamma isomer	0.4
D014	Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0
D015	Toxaphene (C ₁₀ H ₁₀ Cl ₈ , Technical chlorinated camphene, 67-69% chlorine)	0.5
D016	2,4-D (2,4-Dichlorophenoxyacetic acid)	10.0
D017	2,4,5-TP (Silvex) (2,4,5- Trichlorophenoxypropionic acid)	1.0

A) from EPA-SW-846, Second Edition, Section No.2.1.4.

ER-SYM-14: Rev. 4/8

DEPARTMENT USE ONLY

Phone no. _____

DATE PREPARED
DATE REVISED

DEPARTMENT USE ONLY

II. WASTE DESCRIPTION (Must be completed by Generator)

A. General Properties

1. pH range _____ to _____ (based on past analyses or knowledge)
2. Physical state:
 - a. ☐ liquid (less than 20% solids by dry wt. or flowable)
 - b. ☐ gas (ambient temperature and pressure)
 - c. ☐ solid (equal to or greater than 20% by dry wt. and non-flowable)
 - d. ☐ Check here if c. above was checked and waste contains free liquids.
3. Physical appearance:

Color _____ Odor _____

Number of solid or liquid phases of separation _____

Describe each phase of separation _____
4. U.S. DOT proper shipping name UN/NA number, and hazard class (if applicable): _____
5. Typical volume of waste to be shipped to treatment storage or disposal facility:
 - a. Monthly _____ gal., tons (circle one)
 - b. Annually _____ gal., tons (circle one)
6. Treatment or disposal frequency: _____ times per year; ☐ one time
7. Current volume to be shipped to treatment storage or disposal facility _____ gal., tons (circle one)
8.
 - a. Is the waste a hazardous waste as fined in 75.261? ☐ Yes ☐ No
 - b. If yes, describe the hazardous waste according to its description and hazardous waste number in 75.261.
9. Has the waste been delisted as a hazardous waste by DER? ☐ Yes ☐ No ☐ N/A
If yes or N/A, check the appropriate box(es) in Item 10.

MODULE 1, PAGE 3

DATE PREPARED
DATE REVISED

DEPARTMENT USE ONLY

10. Is the waste a residual waste or a delisted hazardous waste? ☐ Yes ☐ No
If yes, check the following box(es) as applicable:

- | | |
|----------------------------------------------------------------|--------------------------------------------------------------------------|
| <input type="checkbox"/> discarded commercial chemical product | <input type="checkbox"/> process waste |
| <input type="checkbox"/> tank bottom | |
| <input type="checkbox"/> off-specification species | <input type="checkbox"/> infectious waste |
| | <input type="checkbox"/> baghouse dust |
| <input type="checkbox"/> manufacturing chemical intermediate | <input type="checkbox"/> wastewater treatment plant residue (industrial) |
| <input type="checkbox"/> still bottom | |
| <input type="checkbox"/> spent catalyst | <input type="checkbox"/> wastewater treatment plant residue (sewage) |
| <input type="checkbox"/> flyash | <input type="checkbox"/> water treatment plant residue |
| <input type="checkbox"/> bottom ash | <input type="checkbox"/> incinerator residue |
| | <input type="checkbox"/> acid mine drainage treatment sludge |
| <input type="checkbox"/> slag | <input type="checkbox"/> spill residue |
| <input type="checkbox"/> foundry sand | |
| <input type="checkbox"/> SO ₂ scrubber sludge | <input type="checkbox"/> other (specify) _____ |

B. Chemical Analyses — *Please attach the following:*

1. The results of the total analysis of the waste as described in the instructions.
2. The results of the leaching tests as described in the instructions and the leaching method.
3. A description of the sampling method.
4. The range of concentrations of the constituents based on knowledge or past analyses.

C. Process Description and Schematic — *Please attach the following:*

1. The substantiation for a confidentiality claim as described in the instructions, if portions of the information you have submitted are confidential.
2. A detailed description of the manufacturing and/or pollution control processes producing the hazardous or residual waste as specified in the instructions.
3. A schematic of the manufacturing and/or pollution control processes producing the hazardous or residual waste as specified in the instructions.

III. Liner Compatibility Evaluation (must be completed by TSD facility)

Attach the results of the liner compatibility evaluation or supporting data as specified in the instructions.

MODULE 1, PAGE 4

DATE PREPARED
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IV. PROPOSED TREATMENT, STORAGE, AND/OR DISPOSAL METHOD (must be completed by TSD facility. Use additional sheets if necessary.)

A. Proposed Treatment Method

B. Proposed Storage Method and Length of Storage

C. Proposed Disposal Method

V. ALTERNATIVES TO PROPOSED TREATMENT AND/OR DISPOSAL METHOD (must be completed by generator. Use additional sheets if necessary.)

A. What Other Treatment, Disposal, Recycle, Reuse, or Reclamation Method(s) Can be Used? Briefly describe viable alternatives to your proposal.

B. Why was the Treatment and/or Disposal Method in Section IV Chosen?

DATE PREPARED
DATE REVISED

FOR DEPARTMENT USE ONLY

VI. CERTIFICATION OF GENERATOR

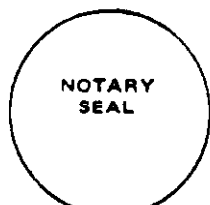
I certify under penalty of law that I have personally examined and am familiar with the information submitted in this and all attached documents, and that based upon my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

Name of Responsible
Official _____

Title _____

Signature _____ Date _____

Taken, sworn and subscribed before me, this



_____ day of _____ A.D. 19 _____

VII. CERTIFICATION OF REGISTERED PROFESSIONAL ENGINEER FOR TREATMENT STORAGE AND/OR DISPOSAL FACILITY

This is to certify that I have personally reviewed all engineering information contained in the accompanying modules, drawings, specifications, and other documents which are part of this application and that I have found it to be of good engineering quality, true and correct, and is in conformance with the requirements of the Department of Environmental Resources, and it does not, to the best of my knowledge, withhold information that is pertinent to a determination of compliance with the requirements of the Department.

NOTICE: It is an offense under Pennsylvania Crimes Code to affirm a false statement in documents submitted to the Department.

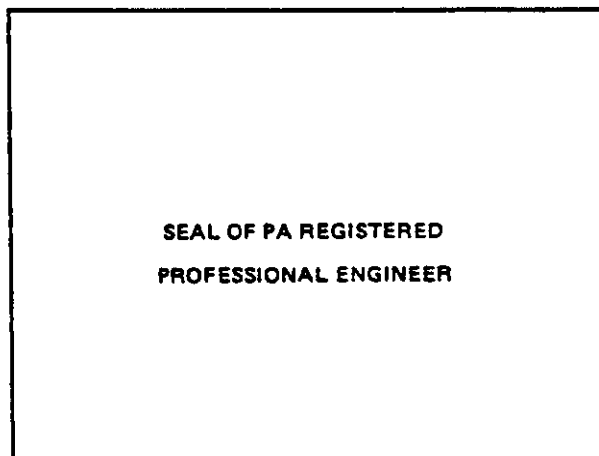
Name _____

Signature _____

Date _____

Address _____

Phone No. _____



INSTRUCTIONS FOR COMPLETING MODULE 1

REQUEST FOR APPROVAL TO TREAT, STORE, OR DISPOSE OF A HAZARDOUS OR RESIDUAL WASTE STREAM (Form ER-SWM-14)

IMPORTANT: Read all instructions carefully before completing module and submit only one waste stream per module.

SECTION I - GENERAL INFORMATION - self explanatory.

SECTION II - WASTE DESCRIPTION

Item A. General Properties

- #3. Describe the color and odor of the waste. (This question is subjective.) For example, gray, solvent odor. Enter number of solid or liquid phases of separation and describe each phase. For example, 2 phases of separation, one solid and one liquid.
- #4. Provide the proper U.S. DOT shipping name, UN/NA number and hazard class for the waste stream. For example, solvents, waste, n.o.s. (toluene, MEK, butanol), UN 1998, flammable.
- #8. a. Check yes if the waste stream is a hazardous waste as described in 75.261.
b. If yes, describe the waste by its characteristic [75.261(g)], the non-specific or specific source lists [75.261(h)(2) and (3)], and/or the commercial chemical product or manufacturing chemical intermediate lists [75.261(h)(4)(v) and (vi)]. List all the reasons which cause the waste to be hazardous. For example, spent pickle liquor from steel finishing operations, K062, Corrosive (D002), and EP toxic (D007).

Item B. Chemical Analyses

These analyses and tests must be performed for all treatment and disposal activities except incineration and thermal treatment. Activities utilizing surface impoundments and waste piles for storage are also required to perform these tests and analyses. Analytical requirements for incineration and thermal treatment can be obtained by contacting the Department.

All analyses submitted must specify the method used and any special preparation, deviation from the method, or pertinent observations. The Method of Standard Addition must be employed to take into account interferences in the matrix of the sample. See EPA's Test Methods for Evaluating Solid Waste (SW-846) or Methods for Analysis of Water and Wastes (EPA-600/4-79-020).

The laboratory performing the analysis must employ the quality control procedures described in EPA's Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA 600/4-79-019). The procedures and documentation of the quality control procedures must be available for inspection if requested by the Department.

- #1. Total Analysis of the Waste - This analysis must include the following list of parameters (a-n) unless the generator certifies in writing the absence of the parameters based on his knowledge of the manufacturing or pollution control processes. Additional parameters, if necessary, must be analyzed to completely characterize the waste. Each analysis sheet must include: date of sampling, date of analysis, name of laboratory performing test, and laboratory contact person and phone number. Report the analyses in mg/kg on a dry weight basis for solids or in mg/l for liquids, or as otherwise specified in cited method.

No single analytical method is applicable for all waste streams and some modifications may be necessary for unusual waste types. Any modification, however, must be approved by the Department.

If the sample is of unknown origin or characteristics contact the Department at (717) 787-7381 prior to analysis.

a. Total Residue

i. Liquid - EPA-600/4-79-020, Method 160.3

ii. Solid - Std. Meth. 14 ED, Method 208 G, pp. 96-98

- b. Total Dissolved Solids - Liquids Only EPA-600/4-79-020, Method 150.1.
- c. Volatile Residue on Total Residue (from a. 1. above)
 - i. Liquid - EPA-600/4-79-020, Method 160.4
 - ii. Solid - Std. Meth. 14 Ed. Method 208 G. pp. 96-98
- d. pH
 - i. Liquid - EPA-600/4-79-020, Method 150.1
 - ii. Solid - 40 CFR Part 250 (Mon. Dec. 18, 1978), Part IV, Appendix IV. Methods for Determining Soil pH, p.59022 reference: "Methods of Soil Analysis" Part II Chemical and Microbiological Properties C. A. Black Ed. (American Society of Agronomy), 1965.
- e. Cyanide - EPA SW-846, Method 8.55
- f. Oil and Grease
 - i. Liquid - EPA-600/4-79-020, Method 413.1
 - ii. Solid - Std. Meth. 14 Ed., Method 502 D, pp. 519-520.
- g. Ammonia-Nitrogen - Liquids Only EPA-600/4-79-020, Method 350.1 or 350.2
- h. Phenolics - Liquids Only EPA-600/4-79-020, Method 420.1 or 420.2
- i. Total Metals - EPA SW-846, General Requirements, Method 8.49, and EPA-600/4-79-020, Metals (AA Methods), Section 200
 - i. Arsenic, EPA SW-846, Method 8.51
 - ii. Barium, EPA, SW-846, Method 8.52
 - iii. Cadmium, EPA SW-846, Method 8.53
 - iv. Chromium, EPA SW-846, Method 8.54
 - v. Lead, EPA SW-846, Method 8.56
 - vi. Mercury, EPA SW-846, Method 8.57
 - vii. Nickel, EPA SW-846, Method 8.58
 - viii. Selenium, EPA SW-846, Method 8.59
 - ix. Silver, EPA SW-846, Method 8.60
 - x. Copper, EPA-600/4-79-020, Method 220.1
 - xi. Molybdenum, EPA-600/4-79-020, Method 246.1
 - xii. Zinc, EPA-600/4-79-020, Method 289.1
- j. Organics - Wastes must be tested for specific solvents, pesticides, or other organic constituents known to be used or produced as a product or byproduct in the process that generated the waste stream. For methods of analysis for specific compounds refer to 75.261 Table A, p.75.68 and EPA's Test Methods for Evaluating Solid Waste (SW-846), or other published procedures. Other methods or modifications may be acceptable if approved by the Department. Contact the Department at (717) 787-7381 for such approval.
- k. Heating Value
 - i. Solid - ASTM Methods D2015, D3826
 - ii. Liquid - ASTM Method D240
- l. Ignitability - Test as described in 75.261 or submit certification that waste is not ignitable
- m. Corrosivity - Test as described in 75.261 or submit certification that waste is not corrosive.

- n. Reactivity - Test as described in 75.261 or submit a statement that waste is not reactive.

#2. Leaching Tests

To demonstrate a waste stream is or is not hazardous by its characteristics, use the EP Toxicity Extraction Procedure 75.261, Appendix II only for parameters listed in 75.261. For codisposal of a waste stream with municipal refuse, the EP Toxicity Extraction Procedure or the ASTM Method B Leaching Procedure. For segregated disposal of a waste stream, either the ASTM or the EP Toxicity Extraction Procedure leachate may be used. The analyses should be conducted on samples in the condition in which they are treated, stored or disposed.

The following constituents and parameters are required in analysis unless they are not present in the total analysis. If the total analysis of the waste indicates less than 0.001 mg/kg or mg/l for a given constituent or parameter, then that constituent or parameter need not be analyzed. Report all results in mg/l or as otherwise specified in method.

- a. pH - EPA-600/4-79-020, Method 150.1 report as pH units
- b. Oil and Grease - EPA-600/4-79-020, Method 413.1
- c. Ammonia-Nitrogen - EPA-600/4-79-020, Method 350.1, or 350.2.
- d. Phenolics - EPA-600/4-79-020, Methods 420.1, or 420.2.
- e. Cyanide - EPA SW-846, Method 8.55.
- f. Total Metals - EPA SW-846, General Requirements, Method 8.49, and EPA-600/4-79-020, Metals (AA Methods), Section 200.
 - i. Antimony, EPA SW-846, Method 8.50
 - ii. Arsenic, EPA SW-846, Method 8.51
 - iii. Barium, EPA SW-846, Method 8.52
 - iv. Cadmium, EPA SW-846, Method 8.53
 - v. Chromium, EPA SW-846, Method 8.54
 - vi. Hexavalent Chromium, EPA 600/4-79-020, Method 8.55
 - vii. Lead, EPA SW-846, Method 8.56
 - viii. Mercury, EPA SW-846, Method 8.57
 - ix. Nickel, EPA SW-846, Method 8.58
 - x. Selenium, EPA SW-846, Method 8.59
 - xi. Silver, EPA SW-846, Method 8.60
 - xii. Copper, EPA-600/4-79-020, Method 220.1
 - xiii. Molybdenum, EPA-600/4-79-020, Method 246.1
 - xiv. Zinc, EPA-600/4-79-020, Method 289.1
- g. Organics - For methods of analysis for specific compounds refer to 75.261 Table A, p.75.68 and EPA's Test Methods for Evaluating Solid Waste (SW-846), or other published procedures. Other methods may be acceptable if approved by the Department.
- h. Total Organic Halogen - Adsorption with microcoulometry detection
- i. COD - EPA-600/4-79-020, Method 410.1, or 410.4
- j. TOC - EPA-600/4-79-020, Method 415.1
- k. Total Volatile Residue - EPA-600/4-79-020, Method 416.1

#3. Description of the sampling method - The procedures outlined in Appendix I of 75.261 must be followed when sampling waste streams.

item C. Process Description and Schematic - Please attach to this module the following:

#1. Confidentiality claim (if any) - Information submitted to the Department in this portion of the module may be claimed as confidential by the applicant. If no claim is made at the time of submission, the Department shall make the information available to the public without further notice.

Claims of confidentiality shall address the following:

- a. The portions of the information claimed to be confidential.
- b. The length of time the information is to remain confidential.
- c. The measures taken to guard against undesired disclosure of the information to others.
- d. The extent the information has been disclosed to others and the precautions taken in connection with that disclosure.
- e. A copy of pertinent confidentiality determinations by EPA or any other federal agency.
- f. The nature of the substantial harm to the competitive position by disclosure of the information, the reasons it should be viewed as substantial and the relationship between the disclosure and the harm.

#2. Describe the manufacturing process which produced the waste and any pollution control methods involved. This must include the raw materials used in the process, any intermediate products formed, final products, and any substances added during treatment. For example:

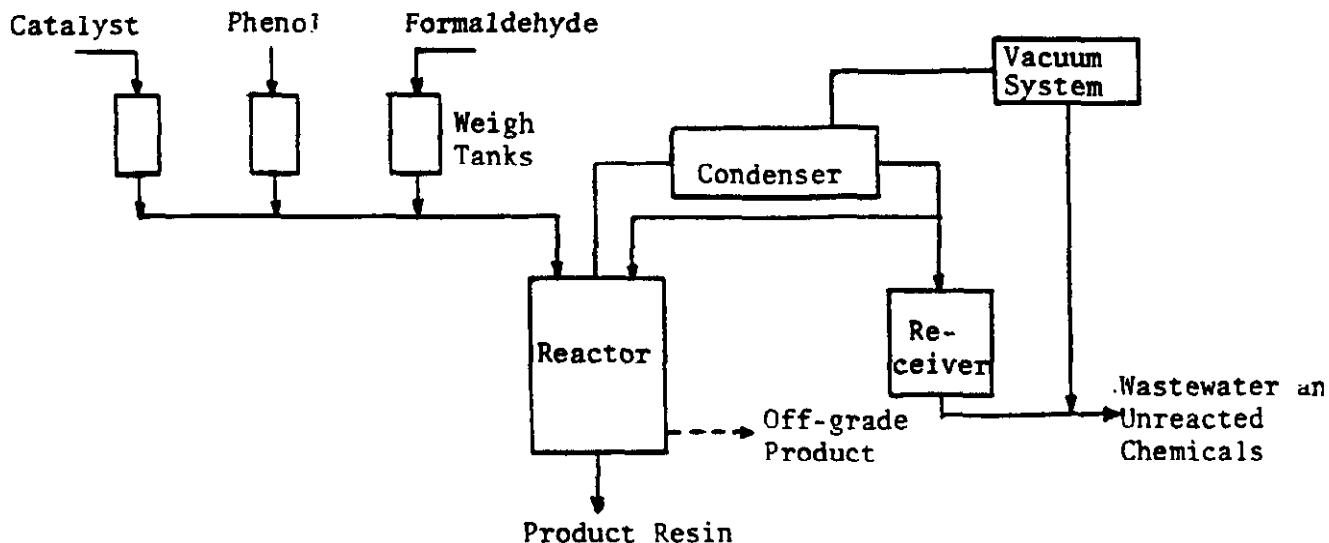
"Resol Resin Manufacture"

"These resins are formed by reacting phenol, or a substituted phenol with formaldehyde which contains an excess of formaldehyde. An alkali (sodium hydroxide) is used to catalyze the polymerization which takes place at a pH of between 8 and 11 and at a temperature of 60°C.

"When the desired degree of polymerization has occurred, the kettle is cooled to about 35°C to inhibit further reaction. The caustic may be neutralized in the kettle with sulfuric acid at this time. The water from this distillation forms a concentrated waste of unreacted materials and low molecular weight resin.

"The batch is dumped, and depending on the specific resin, the batch may be washed several times and a vacuum may be used during the dehydration cycle. It is important that molten resin be handled quickly to avoid its setting up to an insoluble, infusible mass, which would become a waste."

#3. Provide, on 8 1/2" x 11" size paper, flow schematics of the manufacturing and/or pollution control processes generating the hazardous or residual waste stream starting with the raw materials and ending with the final products. For example:



SECTION III - LINER COMPATIBILITY EVALUATION

Liner compatibility testing - any facility utilizing a liner must conduct an evaluation of the liner compatibility with the waste stream before accepting that waste stream for storage, treatment or disposal in a waste pile, surface impoundment, or landfill, unless the approval to accept that waste stream is granted in the facility's permit. The evaluation procedure must be approved by the Department prior to its commencement.

The test protocol will vary with the type of liner system and the characteristics of the waste stream. The Department should be contacted for appropriate test protocols. In lieu of actual testing, existing published or documented data on the hazardous waste or waste generated from similar processes proving the liner compatibility may be substituted in some instances.

SECTION IV - PROPOSED TREATMENT, STORAGE, OR DISPOSAL METHOD

Use additional sheets of paper if necessary.

- item A. Proposed Treatment Method - If applicable, briefly describe the method proposed to treat this waste stream. For example, "Solvent removed from waste by solvent recovery apparatus to less than 1% solvent. Recovered solvent is sold to XYZ, Inc. for reclamation. Solids are polymerized and the remaining solvent is driven off by heat."
- item B. Proposed Storage Method - If applicable, briefly describe the method proposed to store this waste stream and the compatibility with its container, the waste pile liner, or the surface impoundment liner. For example, "Paint waste is placed into 55 gal. steel drums and is proposed to be stored at the XYZ Waste Disposal Company's storage building for 60 days prior to processing. The paint waste is compatible with its container and the other wastes stored in the immediate vicinity. The proposed location for the paint waste within the building is indicated on the attached drawing."
- item C. Proposed Disposal Method - If applicable, briefly describe the method proposed to dispose of this waste stream. For example, "Polymerized solids are to be placed in a segregated cell of the XYZ Waste Disposal Company with compatible wastes as indicated on the attached drawing. The cell is located at coordinates D-7. The cell design has been approved as part of the facility permit."

SECTION V. ALTERNATIVES TO PROPOSED TREATMENT AND/OR DISPOSAL METHOD

- item A. What Other Treatment, Disposal, Recycle, Reuse, or Reclaim Method(s) Can be Used? Briefly describe viable alternatives to your proposal.
- item B. Why was the Treatment and/or Storage Disposal Method in Section III Chosen? Briefly describe why the proposed method was chosen. For example, "The proposed method offers the most cost effective means of disposal over a 10 year period. Capital investment of solvent apparatus and polymerization equipment will be off-set by income from sale of recovered solvent and smaller volumes of waste to be disposed."

SECTION VI. CERTIFICATION OF GENERATOR

The Application Must be Certified in the Following Manner:

- #1. Corporations - A corporate officer must sign the document and the corporate seal must be affixed.
- #2. Limited Partnerships - A general partner must sign the document.
- #3. All other partnerships - A partner must sign the document.
- #4. Sole proprietorships - The proprietor.
- #5. Municipal, state, or federal authority or agency - An executive officer or ranking elected official responsible for compliance of the authority's or agency's hazardous waste activities and facilities with all applicable regulations.

All signatures affixed to the document must be notarized.

SECTION VII. CERTIFICATION OF REGISTERED PROFESSIONAL ENGINEER FOR TREATMENT, STORAGE, OR DISPOSAL FACILITY - Self-Explanatory.

APPENDIX VIII: Letter from Coal Tech Corp. to PA DER,
Bureau of Solid Waste Management, initiating Disposal Permit Application Process.

COAL TECH CORP.

P. O. Box 154
Merion Station, PA. 19066-0154
(215) 667-0442

ATTACHMENT II

April 7, 1987

Mr. Wayne Billings
Bureau of Solid Waste Management
Department of Environmental Resources
Commonwealth of Pennsylvania
200 Pine Street
Williamsport, PA 17701-6510

Dear Mr. Billings:

As per our discussion in your office on March 26, 1987, this letter is a request for written confirmation by DER of our proposed approach to meet the environmental requirements relating to solid waste disposal from Coal Tech's coal combustor tests at the Keeler-Dorr Oliver plant in Williamsport.

Coal Tech is currently engaged in a two year, 3 Phase effort to demonstrate the technology of an advanced coal combustor capable of simultaneous removal of NO_x and SO₂ gas emissions to meet New Source Performance Air Quality Standards.² The combustor will be tested on a 23 million BTU/hr boiler located at the Keeler plant. The project is sponsored in part by the US Department of Energy as part of the "Clean Coal Program". Additional sponsors are: The Pennsylvania Energy Development Authority, and the Pennsylvania Power & Light Company. PP&L is providing 900 tons of PA Freeport Seam coal for the test effort.

SO₂ control is accomplished by injecting limestone in the combustor, where it reacts with the gaseous SO₂. The reacted limestone is then dissolved in the liquid coal ash (slag) lining the combustor wall, and the mixture is drained from the combustor and quenched in a water tank. The solid lime-sulfur-coal slag mixture is to be disposed in an appropriate solid waste disposal site. It is anticipated that 170 tons of solid waste will result from the 900 hours test effort, in the period from mid 1987 through 1988.

This Project is divided into 3 Phases. In Phase 1, which began in February 1987, consists of the design of the test effort and the acquisition of the necessary environmental permits to implement the test effort. Phase 2 consists of test facility modifications and short duration testing at the Keeler plant in Williamsport. The latter is scheduled for the late summer of 1987. Phase 3 is the core of the project and it consists of 900 hours of test which are designed to demonstrate the technology. The 170 tons of solid waste will be generated in Phase 3.

We understand that to dispose of the solid waste, a Module 1 application must be prepared in conjunction with the operator of the disposal site. After approval by DER, the disposal site permit is modified to permit storage of the waste stream.

COAL TECH CORP.

Page 2

April 7, 1987

Anticipating that the tests to be performed on the slag in Phase 2 of the project will result in classifying the slag as a non-hazardous waste, we plan to utilize either the Lycoming landfill or the regional landfill used by PP&L for ash disposal generated by its local power plants. However, we are prepared to utilize other disposal sites, if the Phase 2 test results indicate that an alternate disposal site, (e.g. a hazardous waste site), is required. During Phase 2, the quantity of solid waste generated will be so small that it can be temporarily stored at the Keeler site in several drums.

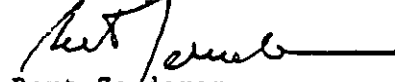
Our confidence in the non-hazardous nature of the slag is based in part on prior tests by Coal Tech on coal slags produced by cyclone combustors, which showed that trace metals did not leach from the slag. With regard to sulfur retention in the slag, the form in which the sulfur is bound in the slag affects its reactivity. This sulfur retention is a function of the combustor's operating method. Therefore, the tests necessary to complete the Module 1 application, cannot be performed before the combustor is operated in Phase 2 of the project. I should note that based on other experimental data on sulfur reactivity in slags, we believe that it will be retained in the slag.

The DOE contract requires written documentation that the permitting process has been initiated in Phase 1 of the project. Therefore, the purpose of this letter is to document that this process has been initiated by contact with your Office and by written confirmation by DER that implementing of the procedure described in this letter will result in a permit for disposal in an appropriate site. The proper site will be dictated by the test results obtained in Phase 2.

In conclusion, a written confirmation by your Office that the above is the proper procedure to be followed for approved solid waste disposal will allow Coal Tech to meet the requirements for Phase 1 of the project. This will enable us to proceed to Phase 2, where the necessary data will be obtained to be used in the Module 1 application.

Thank you for attention to this matter.

Sincerely,


Bert Zauderer,
President

TEST METHOD TO DETERMINE HYDROGEN CYANIDE AND HYDROGEN SULFIDE RELEASED FROM WASTES

1. Scope and Application

- 1.1 This method is applicable to water, wastewater and industrial wastes.
- 1.2 This method provides a fast and simple method to determine if a waste is reactive waste as a result of its tendency to release toxic levels of hydrogen cyanide or hydrogen sulfide upon contact with acidic medium. The approach is based on a reasonable "worst case" disposal scenario.
- 1.3 This method is designed to measure only the hydrogen cyanide and/or hydrogen sulfide gas evolved at the test conditions and not to reflect the total concentration of these gases, or their precursors, in the sample. Variations in temperature, ionic strength and total volume of the test solution will affect the amount of gas evolved. The total volume of solution is kept constant from test to test. Ionic strength is not controlled as it is an inherent property of each waste. In this method, test solutions are not purged with a gas because this would not reflect disposal conditions.

2. Summary of Method

- 2.1 An aliquot of the waste is acidified to pH 2 in a closed system. The gas generated is swept from the reaction chamber using a pump and absorbed in a NaOH solution. The cyanide ion in the absorption solution is quantitated by Method 9010 starting with Step 7.3.5.
- 2.2 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver-sensitive indicator.

3. Sample Handling and Preservation

- 3.1 Samples containing, or suspected of containing sulfide or a combination of sulfide and cyanide wastes, should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and stoppered. Analysis should commence as soon as possible; and samples should be kept in a cool, dark place until analysis begins.
- 3.2 Samples containing or suspected of containing cyanide wastes without the presence of sulfide compounds, can be preserved if analysis cannot begin immediately. Samples are preserved by adjusting them to pH 12 with strong sodium hydroxide solution and storing them in a cool, dark place.

DRAFT

3.3 Determinations should be performed in a well-ventilated hood.

4. Apparatus

- 4.1 Three-neck, round-bottom flask with 24/40 ground-glass joints, 250-ml.
- 4.2 Magnetic stirrer bar with magnetic stirring apparatus.
- 4.3 Separatory funnel with pressure-equilizing tube and 24/40 ground-glass joint, 125-ml.
- 4.4 Straight, glass adapter tubes with 24/40 ground-glass joint and rubber adapter sleeve, 2 each.
- 4.5 Flexible tubing to make connection from detector tube to pump.
- 4.6 Microburet, 5.0 ml (for filtration)
- 4.7 pH meter and pH electrode of sufficient length to reach liquid level (approximately 18 cm).
- 4.8 Pump capable of pulling 60 ± 3 ml per minute (MSA Corporation Model C-210 or equivalent).
- 4.9 Bubble meter for calibrating pump.
- 4.10 Stopwatch.

5. Reagents

- 5.1 Sulfuric Acid, H_2SO_4 1 N.
- 5.2 Cyanide Reference Solution: Dissolve approximately 2.5 g KOH and 2.51 g KCN in one liter of distilled water. Cyanide concentration in this solution is 1 mg/ml.
- 5.3 Sulfide Reference Solution: Prepare from Crystal of sodium sulfide as described in Method 376.2 (Sulfide, Colorimetric, Methylene Blue) Section 5.8 of "Methods for Chemical Analysis of Water and Waste," EPA 600/4-79-020, March, 1979. The solution may be standardized by the titrimetric iodine Method 376.1 of the same publication, although this is not required.
- 5.4 Dilute sodium hydroxide solution, 0.25 N: Dilute 200 mL of sodium hydroxide solution (5.1) to 1 liter with distilled water.
- 5.5 Sodium hydroxide solution, 1.25 N: Dissolve 50 g NaOH in distilled water and dilute to 1 liter with distilled water.

DRAFT

- 5.6 Lead acetate buffer: Dissolve 172.8 gms lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ in 950 ml of water and adjust pH to 4.5 with acetic acid.
- 5.7 Stock cyanide solution, 1 mg/mL: Dissolve 2.51 g KCN and 2 gm KOH in 1 liter of distilled water. Standardized with 0.0192 N AgNO_3 . Dilute to appropriate concentration so that 1 mL = mg CN.
- 5.8 Standard cyanide solution, intermediate 50 mg/L: Dilute 50 mL of stock solution (5.7) to 1 liter with distilled water.
- 5.9 Standard cyanide solution, 5 mg/L: Prepare fresh daily by diluting 100.0 mL of intermediate solution (5.8) to 1 liter with distilled water and store in a glass-stoppered bottle.
- 5.10 Standard silver nitrate solution: Prepare by crushing approximately 5 g of AgNO_3 crystals and drying to constant weight at 40°C . Weigh out 3.2647 g dried AgNO_3 , dissolve in distilled water, and dilute to 1 liter (1 mL = mg CN).
- 5.11 Rhodanine indicator: Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 mL of acetone.
- 5.12 Methyl red indicator: Prepare 0.02 g dissolved in 60 mL water and 40 mL acetic acid.

6. System Check

- 6.1 The operation of the system can be checked using the cyanide or sulfide reference solutions. The reference solutions can be used to verify system operation; and replicated can be expected to agree to within approximately ± 20 percent.

7. Procedure

- 7.1 Place 10 g of the material to be analyzed into a beaker. In a well-ventilated hood, add approximately 80 ml of deionized water and determine the amount of 1 N H_2SO_4 required to adjust the solution pH to 2.0.
- 7.2 Add 500 ml of sodium hydroxide (5.5) to the second (CN) absorbing tube and dilute, if necessary, with distilled water to obtain an adequate depth of liquid.
- 7.3 To the first absorber, (sulfide) add a solution composed of 100 ml of the lead acetate buffer. If necessary make up the volume with distilled water.

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- 7.4 Calibrate the pump in line to a flow of 60 ± 3 ml/min using the bubble meter attached to the pump outlet. Measure the flow before and after the test period.
- 7.5 Place a second 10 g aliquot of the waste into the 250-ml round-bottom flask. Add deionized water in an amount so that the sum of the volume of water and the volume of 1 N H_2SO_4 , required for pH adjustment, will equal 100 ml. Solid samples require a certain amount of dilution to reach a slurry state so that the acid may contact all portions of the sample and so that stirring may take place. Maintain the solution temperature at 30°C .
- 7.6 Assemble the apparatus as shown in Figure 1.
- 7.7 Begin stirring the sample; and make sure all connections are tight.
- 7.8 Maintain the test solution at pH 2.0 using the 1 N H_2SO_4 .
- 7.9 After 30 minutes, stop the pump.
- 7.10 Wash and quantitatively transfer the precipitate in the first scrubber. Determine the sulfide starting with Step 7.2 of Method 9030.
- 7.11 Determine the cyanide in the second absorber starting with Step 7.3.5 of Method 9010.

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8. Calculation

8.1 Using the titrimetric procedure, calculate the concentration of CN as follows:

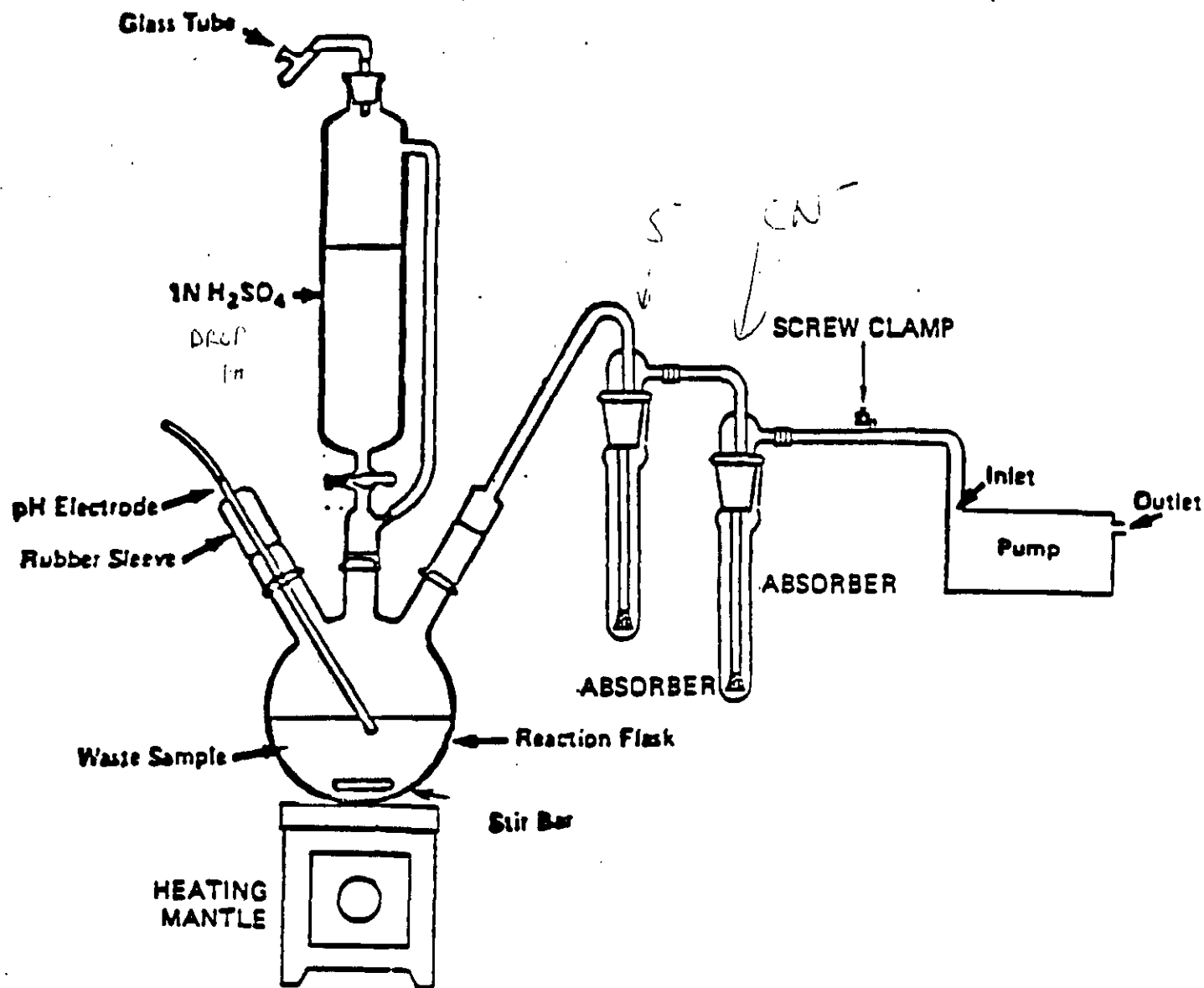
$$\text{CN (mg/L)} = \frac{(A-B)(1000) (100 \text{ mL})}{(\text{mL of original sample}) (\text{mL of aliquot titrated})}$$

where:

A = Volume of AgNO_3 used for titration of sample

A = Volume of AgNO_3 used for titration of blank.

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APPENDIX X.: EP TOXICITY TEST PROCEDURES (a)

2.1.4 Extraction Procedure Toxicity

Introduction

The Extraction Procedure (EP) is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill. This test is designed to simulate leaching that takes place in a sanitary landfill only. It is a laboratory test in which a representative sample of a waste is extracted with distilled water maintained at a pH of 5 using acetic acid. The extract obtained from the EP (the "EP Extract") is then analyzed to determine if any of the thresholds established for the eight elements (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver), four pesticides (Endrin, Lindane, Methoxychlor, Toxaphene), and two herbicides (2,4,5-trichlorophenoxypropionic acid, 2,4-dichlorophenoxyacetic acid) have been exceeded. If the EP Extract contains any one of the above substances in an amount equal to or exceeding the levels specified in 40 CFR 261.24, the waste possesses the characteristic of Extraction Procedure Toxicity and is a hazardous waste.

Summary of Procedure

The Extraction Procedure consists of five steps (refer to Figure 1):

1. Separation Procedure

A waste containing unbound liquid is filtered and if the solid phase is less than 0.5% of the waste, the solid phase is discarded and the filtrate analyzed for trace elements, pesticides, and herbicides (step 5). If the waste contains more than 0.5% solids, the solid phase is extracted and the liquid phase stored for later use.

2. Structural Integrity Procedure/Particle Size Reduction

Prior to extraction, the solid material must pass through a 9.5-mm (0.375-in.) standard sieve, have a surface area per gram of waste of 3.1 cm², or, if it consists of a single piece, be subjected to the Structural Integrity Procedure. The Structural Integrity Procedure is used to demonstrate the ability of the waste to remain intact after disposal. If the waste does not meet one of these conditions it must be ground to pass the 9.5-mm sieve.

3. Extraction of Solid Material

The solid material from step 2 is extracted for 24 hr in an aqueous medium whose pH is maintained at or below 5 using 0.5 N acetic acid. The pH is maintained either automatically or manually. (In acidifying to pH 5, no more than 4.0 g of acid solution per g of material being extracted may be used.)

a) From EPA-SW-846, Second Edition, Section 4.1.4

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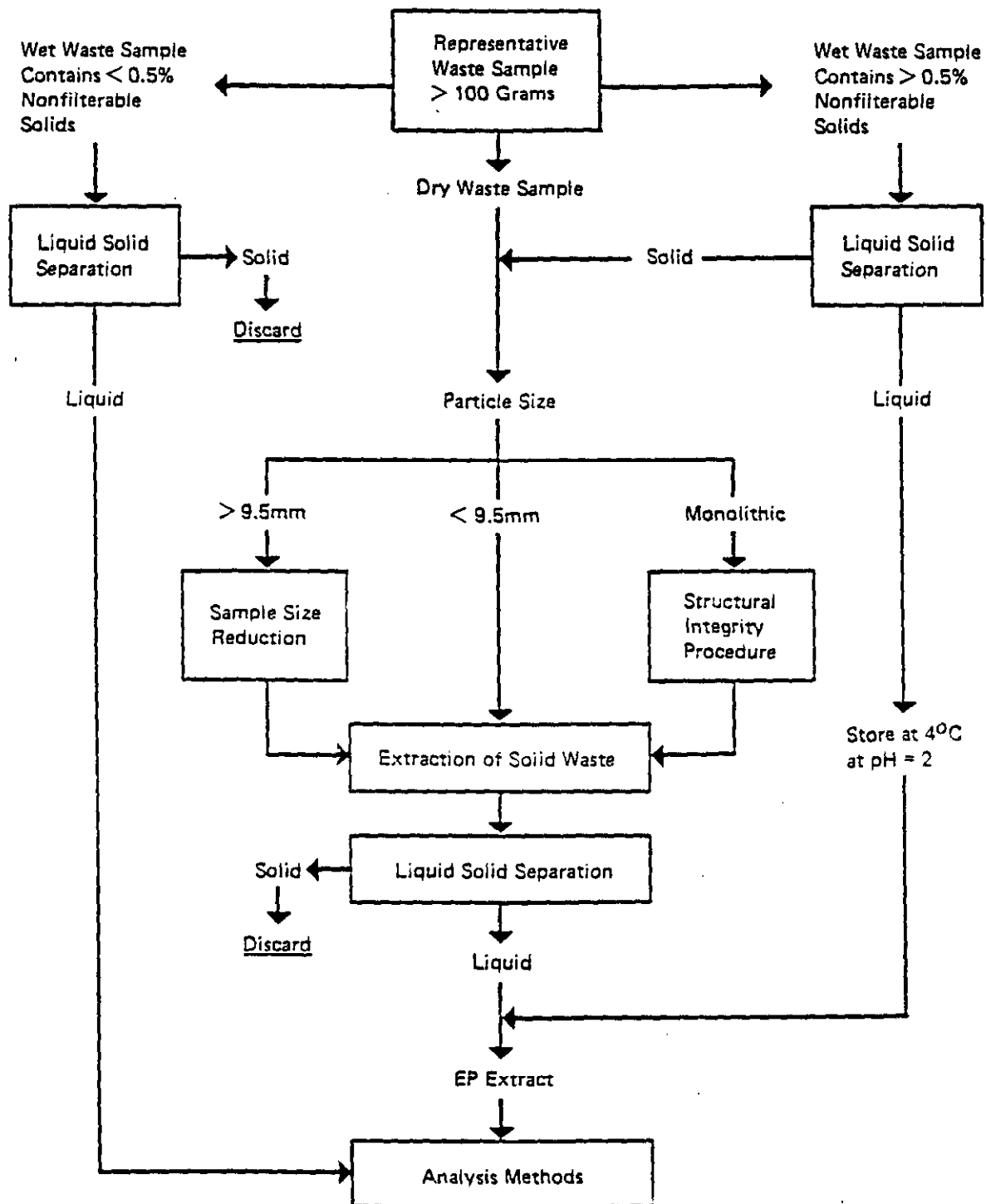


Figure 1. Extraction Procedure Flowchart.

4. Final Separation of the Extraction from the Remaining Solid

After extraction, the liquid:solid ratio is adjusted to 20:1 and the mixed solid and extraction liquid are separated by filtration. the solid is discarded and the liquid combined with any filtrate obtained in step 1. This is the EP Extract that is analyzed and compared to the threshold listed in Table 1 of 40 CFR 261.24.

5. Testing (Analysis) of EP Extract

Inorganic and organic species are identified and quantified using the appropriate methods in the 7000 and 8000 series of methods in this manual.

Regulatory Definition

A solid waste exhibits the characteristic of EP toxicity if, using the appropriate test methods described in this manual or equivalent methods approved by the Administrator under the procedures set forth in 40 CFR 260.20 and 260.21, the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 at a concentration equal to or greater than the respective value given in that Table. If a waste contains less than 0.5% filterable solids, the waste itself, after filtering, is considered to be the extract for the purposes of analysis.

A solid waste that exhibits the characteristic of EP toxicity, but is not listed as a hazardous waste in Subpart D, is assigned EPA Hazardous Waste Numbers that correspond to the toxic contaminants causing it to be hazardous. These numbers are specified in Table 1.

METHOD 1310

EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD AND STRUCTURAL INTEGRITY TEST

1.0 Scope and Application

1.1 The extraction procedure (EP) described in this method is designed to simulate the leaching a waste will undergo if disposed of in an improperly designed sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphasic samples.

2.0 Summary of Method

2.1 If a representative sample of the waste contains more than 0.5% solids, the solid phase of the sample is extracted with deionized water which is maintained at a pH of 5 ± 0.2 using acetic acid. The extract is analyzed to determine if any of the threshold limits listed in Table 1 are exceeded. Table 1 also specifies the approved method of analysis. Wastes that contain less than 0.5% solids are not subjected to extraction, but are directly analyzed and evaluated in a manner identical to that of extracts.

3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods referenced in Table 1.

4.0 Apparatus and Materials

4.1 Extractor: For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures 1-3 of this method and Section 2.2 (Mobility) of this manual and are available from Associated Designs & Manufacturing Co., Alexandria, Virginia; Kraft Apparatus Inc., Mineola, New York; Millipore, Bedford, Massachusetts; and Rexnord, Milwaukee, Wisconsin.

4.2 pH Meter or pH Controller (Chemtrix, Inc., Hillsboro, Oregon is a possible source of a pH controller).

4.3 Filter holder: A filter holder capable of supporting a 0.45- μ filter membrane and able to withstand the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 5.3 kg/cm² (75 psi) of pressure. The type of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table 2.

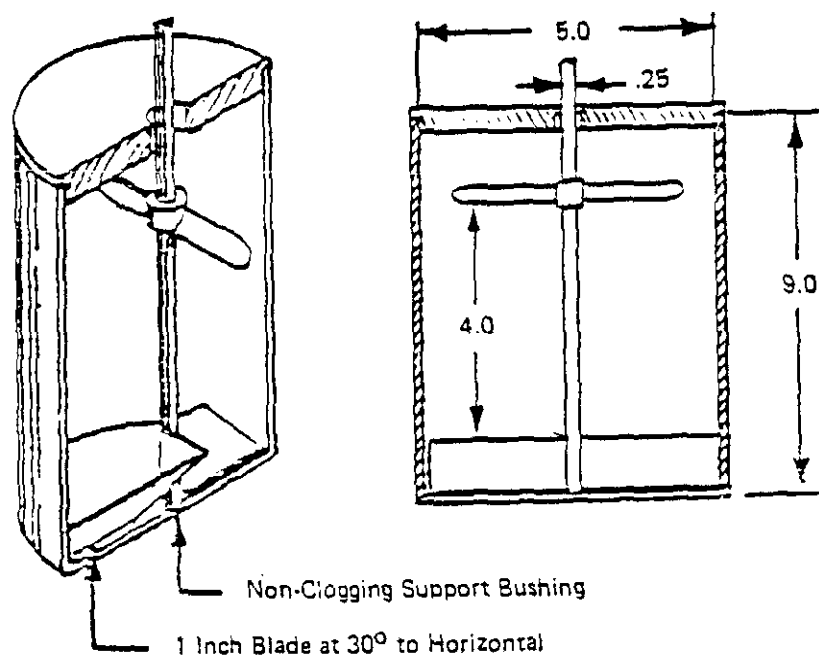


Figure 1. Extractor.

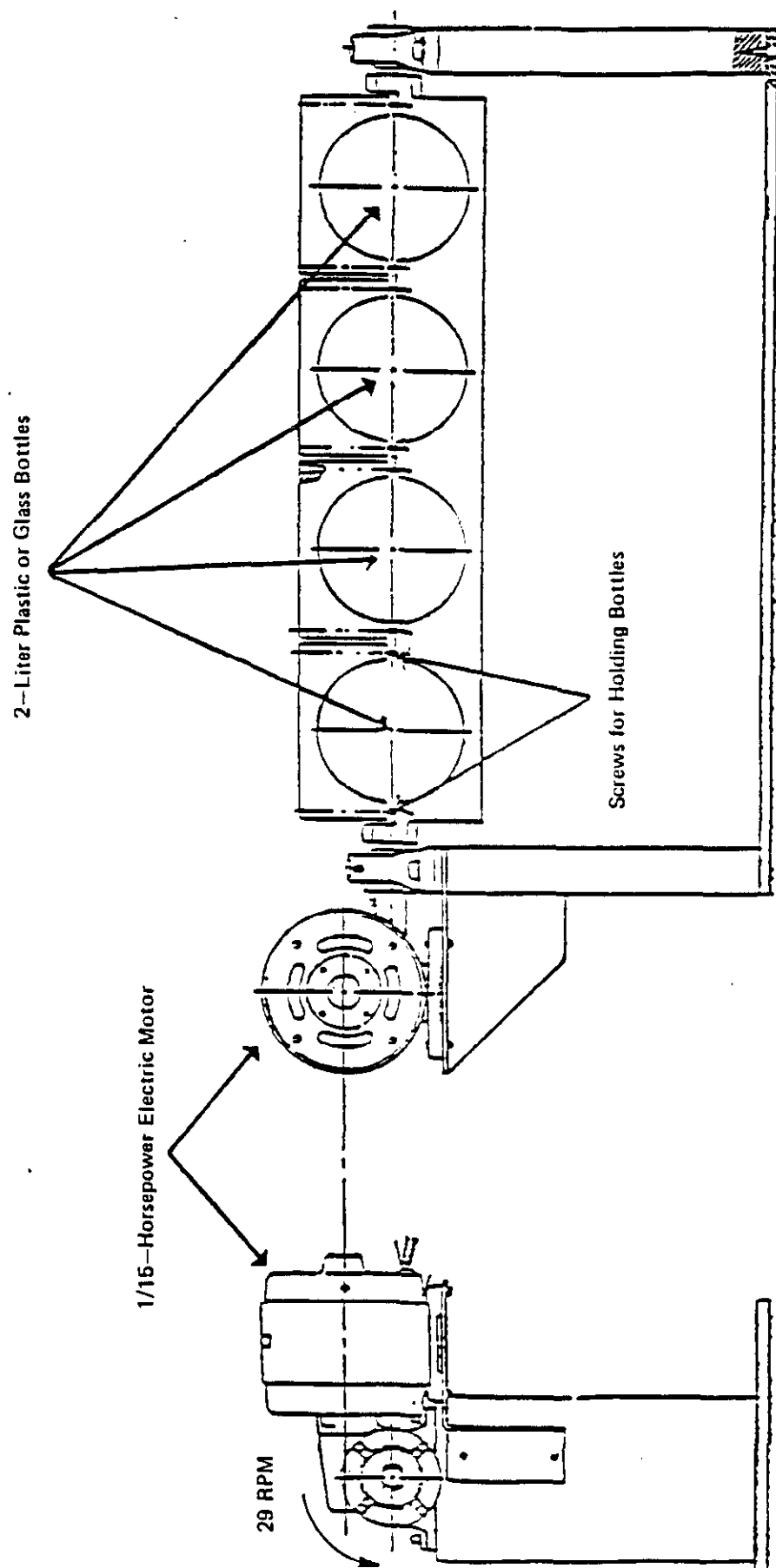


Figure 2. Rotary Extractor.

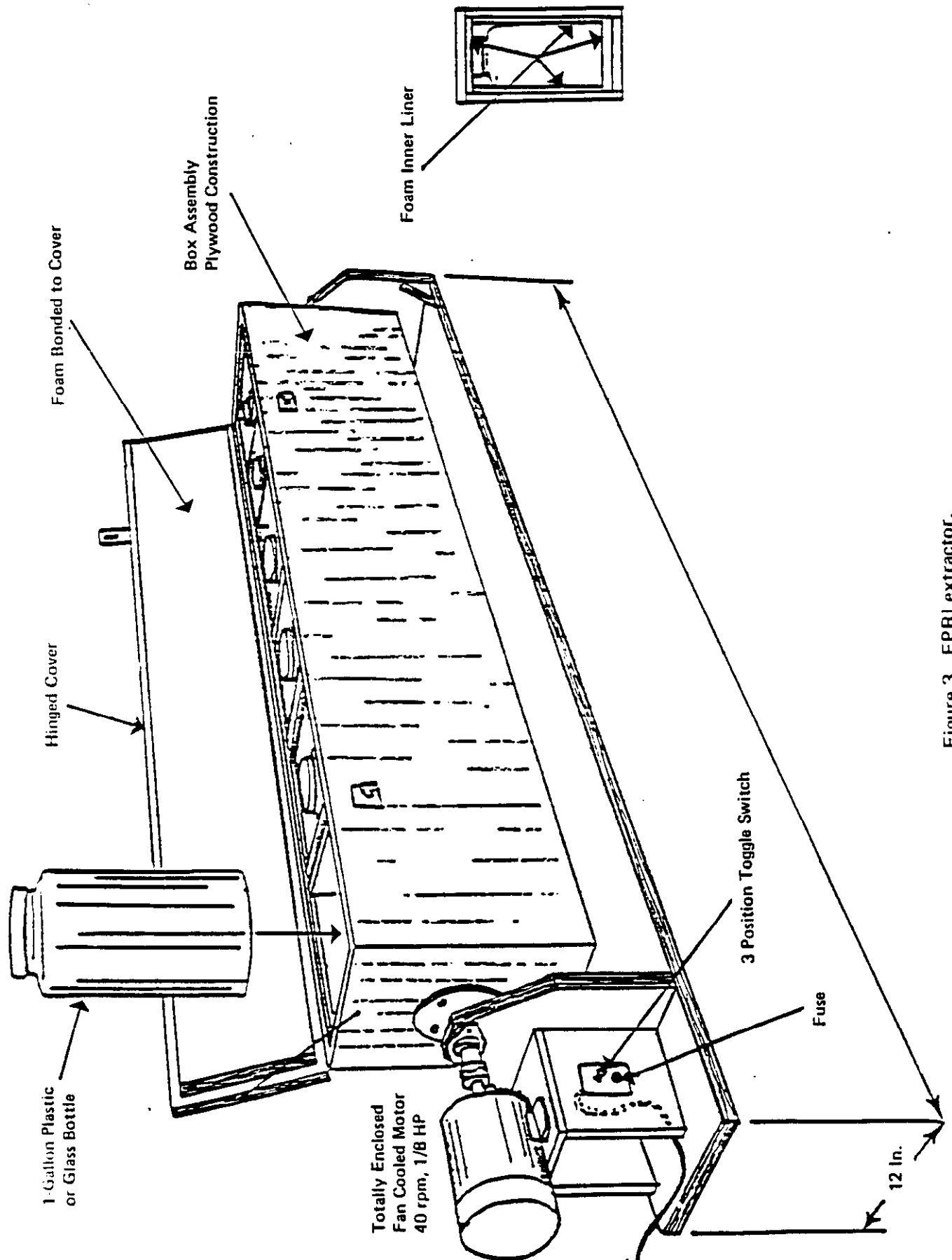


Figure 3. EPR1 extractor.

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4.4 Filter membrane: Filter membrane suitable for conducting the required filtration shall be fabricated from a material which: (1) is not physically changed by the waste material to be filtered, and (2) does not absorb or leach the chemical species for which a waste's EP Extract will be analyzed. Table 3 lists filter media known to the agency and generally found to be suitable for solid waste testing.

4.4.1 In cases of doubt, contact the filter manufacturer to determine if the membrane or the prefilter are adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. After 48 hr, a filter that undergoes visible physical change (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

TABLE 2. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
<u>Vacuum Filters</u>			
Nalgene	500 ml	44-0045	Disposable plastic unit, includes prefilter and filter pads, and reservoir; should be used when solution is to be analyzed for inorganic constituents
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
<u>Pressure Filters</u>			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

TABLE 3. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
<u>Coarse Prefilter</u>		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
<u>Medium prefilters</u>		
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
<u>Fine prefilters</u>		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
<u>Fine filters (0.45 μm)</u>		
Gelman	60173, 60177	60540 or 66149, 60544 or 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218 ^a
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

^aSusceptible to decomposition by certain polar organic solvents.

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4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and re-analyze. If the concentration of the filtrate differs from the original standard, the filter membrane leaches or absorbs one or more of the chemical species.

4.5 Structural integrity tester: Having a 3.18-cm (1.25-in.) diameter hammer weighing 0.33 kg (0.73 lb) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 4.

5.0 Reagents

5.1 Deionized water: Water should be monitored for impurities.

5.2 0.5 N acetic acid: This can be made by diluting concentrated glacial acetic acid (17.5 N). The glacial acetic acid should be of high purity and monitored for impurities.

5.3 Analytical standards should be prepared according to the analytical methods referenced in Table 1.

6.0 Sample Collection, Preservation and Handling

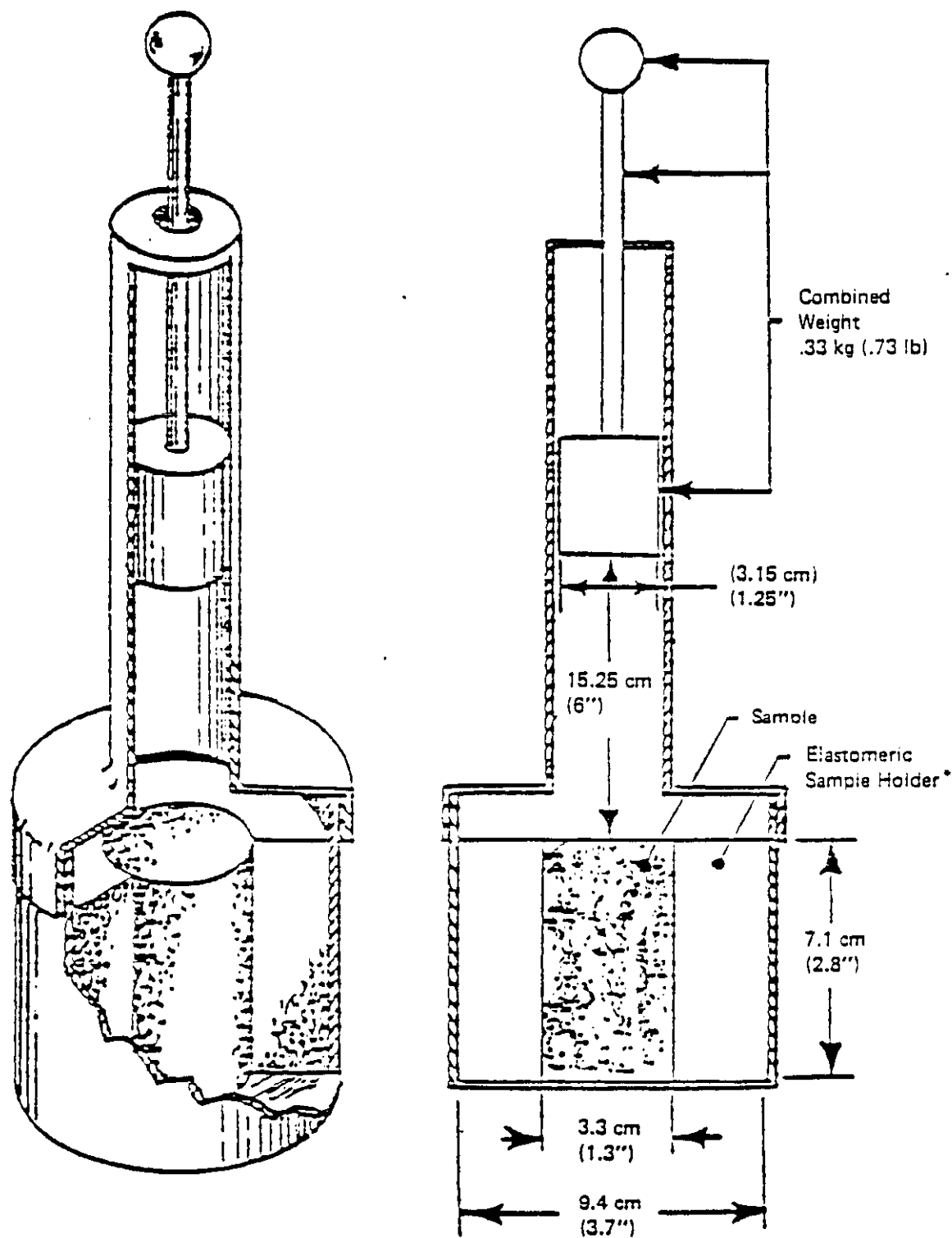
6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 Preservatives must not be added to samples.

6.3 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

7.0 Procedure

7.1 If the waste does not contain any free liquid, go to Section 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to ± 0.01 g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipette.



* Elastomeric sample holder fabricated of material firm enough to support the sample.

Figure 4. Compoaction tester.

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7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45- μ m membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100 g minimum).

7.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Section 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples which contain oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not filter (this is usually the case with heavy oils or greases) it is carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing it to dry, weight to ± 0.01 g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Section 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on depending on whether it contains more or less than 0.5% solids. If the sample appears to have less than 0.5% solids, the percent solids will be determined by the following procedure.

7.7.1 Dry the filter and residue at 80° C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids using the following equation:

$$\frac{\text{weight of filtered solid and filters} - \text{tared weight of filters}}{\text{initial weight of waste material}} \times 100 = \% \text{ solids}$$

NOTE: This procedure is only used to determine whether the solid must be extracted or whether it can be discarded unextracted. It

is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80° C. A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid comprises less than 0.5% of the waste, discard the solid and proceed immediately to Section 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Section 7.5 and all materials that do not contain free liquids should be evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm² or passes through a 9.5-mm (0.375-in.) standard sieve, the operator should proceed to Section 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material would be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5-mm (0.375-in.) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Section 7.10.

7.10 Structural Integrity Procedure (SIP):

7.10.1 Cut a 3.3-cm-diameter by 7.1-cm-long cylinder from the waste material. For wastes that have been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the Extractor.

7.11 If the sample contains more than 0.5% solids, use the wet weight of the solid phase obtained in Section 7.6 for purposes of calculating the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_f - W_t$$

where:

W = wet weight in grams of solid to be charged to extractor

W_f = wet weight in grams of filtered solids and filter media

W_t = weight in grams of tared filters.

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If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Section 7.11) into the extractor and add 16 times its weight of deionized water.

7.13 After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and, if the pH rises above 5.2, 0.5 N acetic acid should be added to bring the pH down to 5.0 ± 0.2 . However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hr and maintained at $20^{\circ}\text{--}40^{\circ}\text{ C}$ ($68^{\circ}\text{--}104^{\circ}\text{ F}$) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5 N acetic acid. If such a system is not available, the following manual procedure shall be employed.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2 . The pH of the solution should be adjusted at 15-, 30-, and 60-min intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5 pH units.

7.13.3 The adjustment procedure should be continued for at least 6 hr.

7.13.4 If, at the end of the 24-hr extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional 4 hr, during which the pH should be adjusted at 1-hr intervals.

7.14 At the end of the extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where:

V = ml deionized water to be added

W = weight in g of solid charged to extractor

A = ml of 0.5 N acetic acid added during extraction

7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner.

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Section 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10 psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Sections 7.5 and 7.15 should be combined. This combined liquid (or the waste itself if it has less than 0.5% solids, as noted in Section 7.8) is the extract and should be analyzed for the presence of any of the contaminants specified in Table 1 using the Analytical Procedures designated in Section 7.17.

7.17 The extract will be prepared and analyzed according to the analytical methods specified in Table 1. All of these analytical methods are included in this manual. The method of standard addition will be employed for all metal analyses.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 ml of oil and 1,000 ml of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be

$$\frac{(50)(\text{contaminant conc. in oil})}{1,050} + \frac{(1,000)(\text{contaminant conc. of aqueous phase})}{1,050}$$

7.18 The extract concentrations are compared to the maximum contamination limits listed in Table 1. If the extract concentrations are equal to or greater than the respective values, then the waste is considered to be EP toxic.¹

¹Chromium concentrations have to be interpreted differently. A waste containing chromium will be determined to be EP toxic if (1) the waste extract has an initial pH of less than 7 and contains more than 5 mg/l of hexavalent chromium in the resulting extract, or (2) the waste extract has an initial pH greater than 7 and a final pH greater than 7 and contains more than 5 mg/l of hexavalent chromium in the extract, or (3) the waste extract has an initial pH greater than 7 and a final pH less than 7 and contains more than 5 mg/l of total chromium, unless the chromium is trivalent. To determine whether the chromium is trivalent, the sample must be processed according to an alkaline digestion method (Method 3060) and analyzed for hexavalent chromium (Methods 7195, 7196, or 7197).

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8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 All quality control measures suggested in the referenced analytical methods should be followed.

APPENDIX XI.: METHOD 9010 FOR CYANIDE IN WATER
DETERMINATION OF TEST METHODS FOR
EVALUATING SOLID WASTE
PHYSICAL/CHEMICAL METHODS

SW-846

2nd Edition

U.S. ENVIRONMENTAL PROTECTION AGENCY
1982

METHOD 9010

TOTAL AND AMENABLE CYANIDE

1.0 Scope and Application

1.1 Method 9010 is used to determine the concentration of inorganic cyanide in a waste or leachate. The method detects inorganic cyanides that are present as either simple soluble salts or complex radicals. It is used to determine values for both total cyanide and cyanide amenable to chlorination. Method 9010 does not determine the "reactive" cyanide content of wastes containing iron-cyanide complexes. (As an alternative to Method 9010, autoanalyzers may be used for cyanide analysis if the analyst adheres to the precautions and quality control requirements specified in this method.)

2.0 Summary of Method

2.1 The waste is divided into two parts. One is chlorinated to destroy susceptible complexes. Each part is then distilled to remove interferences and analyzed for cyanide. The fraction amenable to chlorination is determined by the difference in values.

2.2 During the distillation, cyanide is converted to hydrogen cyanide vapor, which is trapped in a scrubber containing sodium hydroxide solution. This solution is then titrated with standard silver nitrate.

3.0 Interferences

3.1 Sulfides interfere with the titration. They may be precipitated with cadmium.

3.2 Fatty acids form soaps under alkaline titration conditions and interfere. They may be extracted with a suitable solvent.

3.3 Oxidizing agents may decompose the cyanide. They may be treated with ascorbic acid.

3.4 Thiocyanate presence will interfere by distilling over in the procedure. This can be prevented by adding magnesium chloride.

3.5 Aldehydes and ketones may convert cyanide to cyanohydrin under the acid distillation conditions.

4.0 Apparatus and Materials

4.1 Microburet, 5.0 ml, for titration.

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4.2 Flasks, condenser, and tubing are needed as shown in Figure 1. The boiling flask should be of 1-liter size with inlet tube and provision for a condenser. The gas absorber may be a Fisher-Milligan scrubber. Assemble as shown in Figure 1.

5.0. Reagents

5.1 ASTM Type II water (ASTM D1193) or better quality: Water should be monitored for impurities.

5.2 Calcium hypochlorite solution: Dissolve 5 g of hypochlorite, $\text{Ca}(\text{OCl})_2$, in 100 ml of Type II water.

5.3 Sodium hydroxide solution (1.25 N): Dissolve 50 g of sodium hydroxide (NaOH) in Type II water and dilute to 1 liter.

5.4 Ascorbic acid: crystals.

5.5 Potassium iodide-starch paper.

5.6 Lead acetate paper.

5.7 Cadmium carbonate (powdered).

5.8 Hexane.

5.9 Acetic acid solution (1:9).

5.10 Conc. H_2SO_4 .

5.11 Silver nitrate standard solution (0.0192 N): Dry 5 g AgNO_3 crystals to constant weight at 40°C . Weigh out 3.2647 g and dissolve in Type II water. Dilute 1000 ml (1 ml = 1 mg CN).

5.12 Rhodanine indicator solution: Dissolve 20 mg p-dimethyl-amino-benzalrhodanine in 100 ml acetone.

5.13 Magnesium chloride solution: Weigh 510 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ into a 1-liter volumetric flask. Dissolve and bring to volume with Type II water.

6.0 Sample Collection, Preservation and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 Samples should be collected in plastic or glass bottles of 1-liter size or larger. All bottles must be thoroughly cleaned and thoroughly rinsed to remove soluble materials from containers.

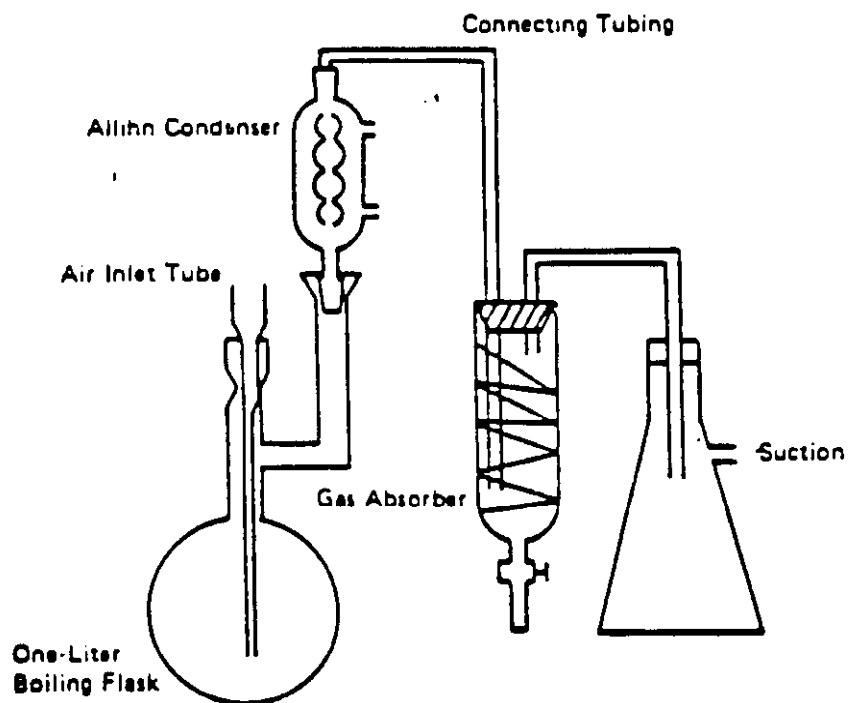


Figure 1. Apparatus for cyanide distillation.

4 / MISCELLANEOUS ANALYTICAL METHODS

6.3 Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper; a blue color indicates the need for treatment. Add ascorbic acid a few crystals at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of water.

6.4 Samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH is greater than or equal to 12) at the time of collection.

6.5 Samples should be refrigerated at 4° C when possible and analyzed as soon as possible.

7.0 Procedure

7.1 If interferences are known or suspected to be present, test and treat the sample as follows.

7.1.1 Sulfides: If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 ml more of the sample than the amount required for the cyanide determination with powdered calcium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to use for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide.

7.1.2 Fatty acids: Acidify the sample with acetic acid (1:9) to pH 6.0 to 7.0. CAUTION: Toxic hydrogen cyanide can be generated in an acid solution. This operation must be performed in the hood and the sample left there until it can be made alkaline again after the extraction has been performed. Then extract with isooctane, hexane, or chloroform (preference in order listed) with a solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the fatty acids below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with NaOH solution.

7.1.3 Oxidizing agents: Test a drop of the sample with potassium iodide-starch test paper (KI-starch paper). A blue color indicates the need for treatment. Add ascorbic acid a few crystals at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

7.2 Chlorination of a sample aliquot

7.2.1 Take a 500-ml sample aliquot or a sample volume diluted to 500 ml. Add calcium hypochlorite solution dropwise while agitating and maintaining the pH between 11 and 12 with sodium hydroxide solution (1.25 N). CAUTION: The initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride; therefore, this reaction should be performed in a hood. For convenience, the sample may be agitated in a 1-liter beaker by a magnetic stirring device.

7.2.2 Test for residual chlorine with KI-starch paper and maintain this excess for 1 hr, continuing agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional hypochlorite solution.

7.2.3 After 1 hr, add 0.5-g portions of ascorbic acid until KI-starch paper shows no residual chlorine. Add an additional 0.5 g of ascorbic acid to ensure the presence of excess reducing agent.

7.3 Take the aliquot treated in Section 7.2 plus either a second 500-ml aliquot of the untreated sample or an untreated aliquot diluted to 500 ml in the 1-liter boiling flask and separately distill as follows.

7.3.1 Add 50 ml of sodium hydroxide (1.25 N) to the absorbing tube and dilute if necessary with Type II water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber, and trap in the train.

7.3.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube. CAUTION: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. The air rate must therefore occasionally be adjusted to prevent the solution in the boiling flask from backing up into the air inlet tube.

7.3.3 Slowly add 25 ml conc. sulfuric acid through the air inlet tube. Rinse the tube with Type II water and allow the air flow to mix the flask contents for 3 min. Pour 20 ml of magnesium chloride solution into the air inlet and wash down with a stream of water.

7.3.4 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for 1 hr. Turn off the heat and continue the airflow for at least 15 min. After cooling the boiling flask, disconnect the absorber and close off the vacuum source.

6 / MISCELLANEOUS ANALYTICAL METHODS

7.3.5 Drain the solution from the absorber into a 250-ml volumetric flask and bring up to volume with Type II water washings from the absorber tube.

7.4 Titration

7.4.1 Add the solution or an aliquot diluted to 250 ml to a 500-ml erlenmeyer flask. Add 10-12 drops rhodanine indicator.

7.4.2 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a Type II water blank using the same amount of sodium hydroxide and indicator as in the sample.

7.4.3 The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5- or 10-ml microburet may be conveniently used to obtain precise titration.

7.5 Titrate a blank using Type II water in an identical manner.

7.6 Calculation:

$$1. \text{ CN, mg/l} = \frac{(A - B)1,000}{\text{ml orig. sample}} \times \frac{250}{\text{ml of aliquot titrated}}$$

where:

A = volume of AgNO_3 for titration of sample.

B = volume of AgNO_3 for titration of blank.

2. Cyanide amenable to chlorination:

$$\text{CN, mg/l} = C - D$$

where:

C = mg/l total cyanide in unchlorinated aliquot

D = mg/l total cyanide in chlorinated aliquot

7.7 Duplicates, spiked standards, and check standards should be routinely analyzed.

8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1633a

Trace Elements in Coal Fly Ash

This Standard Reference Material (SRM) is intended for use in the evaluation of analytical methods for the determination of constituent elements in coal fly ash or materials with a similar matrix.

SRM 1633a is a fly ash that was sieved through a No. 170 sieve with a nominal sieve opening of 90 μm .

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the analysts are given in Table 3. The certified values are based on results obtained by reference methods of known accuracy or from two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This certification is invalid 5 years from date of purchase of the SRM. The constituents certified or analyzed are reviewed periodically and may be updated to reflect improved measurement. Updated certificates will be made available upon request.

Use: This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) Vacuum drying for 24 hours at ambient temperature using a cold trap at or below -50°C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 2 hours in an oven at 105°C ; (3) drying in a dessicator over P_2O_5 or Mg_2ClO_4 . Samples of the dried material weighing at least 250-mg should be used for analysis. When not in use the material should be kept in a tightly sealed bottle.

Source and Preparation of Material: The fly ash material was supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. It was selected as a typical fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was sieved and blended for 2 hours in a Vee blender. The material was then removed and placed in a series of bulk containers from which specific samples were taken for homogeneity testing and certification analysis. Twelve bottles were selected for the homogeneity test. Samples from each bottle were analyzed for cobalt, chromium, europium, iron, scandium, and thorium using nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg sample sizes were consistent with counting statistics, indicating that the fly ash is homogeneous within $\pm 5\%$ (relative) based on these elements. The homogeneity testing and certification analyses were performed in the NBS Center for Analytical Chemistry.

The overall direction and coordination of the analytical measurements leading to the initial certification were performed in the Center for Analytical Chemistry under the chairmanship of L.A. Machlan.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W.P. Reed and T.E. Gills.

Gaithersburg, MD 20899
January 5, 1985
(Revision of certificate
dated April 18, 1979)

Stanley D. Raspberry, Chief
Office of Standard Reference Materials

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Table 1. Certified Values of Constituent Elements

<u>Major Constituents</u>	<u>Content Wt. Percent</u>		<u>Minor Constituents</u>	<u>Content Wt. Percent</u>	
Aluminum	14.3 ± 1.0 ^a	27.0	Magnesium	0.455 ± 0.010	0.75
Iron	9.4 ± 0.1	13.4	Sodium	0.17 ± 0.01	0.23
Potassium	1.88 ± 0.06	2.26			
Silicon	22.8 ± 0.8	48.7 ⁷			
Calcium	1.11 ± 0.01	1.55			

Trace Constituents

<u>Element</u>	<u>Content µg/g</u>	<u>Element</u>	<u>Content µg/g</u>
Antimony	6.8 ± 0.4	Rubidium	131 ± 2
Arsenic	145 ± 15	Selenium	10.3 ± 0.6
Cadmium	1.00 ± 0.15	Strontium	830 ± 30
Chromium	196 ± 6	Thorium	24.7 ± 0.3
Copper	118 ± 3	Thallium	5.7 ± 0.2
Manganese	179 ± 8	Uranium	10.2 ± 0.1
Mercury	0.16 ± 0.01	Vanadium	297 ± 6
Nickel	127 ± 4	Zinc	220 ± 10
Lead	72.4 ± 0.4		

^aThe uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents).

Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

Table 2. Noncertified Values for Constituent Elements

<u>Element</u>	<u>Content Wt. Percent</u>	<u>Element</u>	<u>Content µg/g</u>
Barium	0.15	Beryllium	12
Titanium	0.8 1.3 ^a	Cerium	180
Sulfur	0.18	Cobalt	46
		Cesium	11
		Europium	4
		Gallium	58
		Hafnium	8
		Molybdenum	29
		Scandium	40

Table 3. Analytical Methods Used for Certified Constituent Elements

Method/ Element	A	B	C	D	E	F	G	H	I
Aluminum	.		.						.
Antimony			.				.		
Arsenic	.		.						
Cadmium			
Calcium	.	.			.				
Chromium	.	.	.						
Copper	.	.	.						
Iron	.	.	.						
Lead		.		.	.				
Magnesium	.	.							
Manganese	.		.						.
Mercury	.		.						
Nickel				
Potassium	.	.			.				
Rubidium				
Selenium	.		.				.		
Silicon					.			.	
Sodium	.		.						
Strontium	.				.	.			
Thallium		.					.		
Thorium		.	.						
Uranium		.							
Vanadium	.	.	.						
Zinc			

Analytical Methods

- A. Atomic Absorption Spectrometry or Flame Emission Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. X-ray Fluorescence Spectrometry
- F. Inductively-Coupled Plasma Emission Spectrometry
- G. Isotope Dilution Spark Source Mass Spectrometry
- H. Gravimetry
- I. Direct Coupled Plasma Emission Spectrometry

National Bureau of Standards

Certificate of Analysis

Standard Reference Materials

2689, 2690, and 2691

Coal Fly Ashes

These Standard Reference Materials (SRM's) are intended for use in the evaluation of analytical methods and techniques used for the classification of coal fly ash and for the determination of constituent elements in coal fly ash or materials with a similar matrix. Each SRM consists of 3 hermetically sealed glass vials of fly ash, which has been size classified and blended to a high degree of homogeneity. Each vial contains 10 grams of fly ash.

The three different fly ash SRM's were chosen to cover a wide range of chemical and mineralogical compositions. The certified values for the constituent elements and the sieve residue are given in Table 1. For user convenience, gravimetric multipliers for the conversion of the constituent elements to oxides are given in Table 2. The certified values, except for the sieve residue, are based on measurements using two or more independent reliable analytical techniques and/or methods. Noncertified values (in parentheses), also given in Table 1, are provided for information only.

The certified value for the residue retained on a No. 325 U.S. Standard Sieve is theoretical and is based on an ideal 45 micrometer sieve. The theoretical value was calculated from a least-squares straight line fit of mean values of measurements made using NBS calibrated sieves with average sieve openings of 42.5, 44.0, 45.5, 46.0, 46.5, and 47.0 micrometers. The residue data were obtained from tests performed in accordance with ASTM C430, Standard Test Method for Fineness of Hydraulic Cement by the 45 micrometer (No. 325) Sieve, as specified in ASTM C311, Standard Methods of Sampling and Testing Fly Ash or Natural Pozzolans for Use as Mineral Admixture in Portland Cement Concrete.

Information on the sources of the fly ashes and a description of the coals from which they were derived is given in Table 3. A list of analytical techniques and methods that were used for the certification of these SRM's is given in Table 4.

Notice to Users: These SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used.

Use: Before certification, these fly ash SRM's were homogenized and hermetically sealed in glass vials to minimize any changes in chemical and physical properties. Therefore, the SRM's should be used as received.

To open a vial containing the SRM, make a deep scratch with a file 1/4 inch from the bottom. Invert the vial and press a red hot file point against the scratch to cause a circumferential crack to form.

The composition and specific surface area of the ash may change on being exposed to the moisture in the air. Therefore, the SRM should be used as soon as possible after opening. If not used immediately, it should be protected from atmospheric moisture by transferring the opened SRM to a stoppered vial and storing in a desiccator.

The preparation of these SRM's and the coordination of cooperative technical measurements leading to certification were performed under the direction of Howard M. Kanare with assistance from Charles M. Wilk, both of Construction Technology Laboratories, Portland Cement Association, Skokie, Illinois.

The overall direction and coordination of the analytical measurements leading to certification were performed in the Inorganic Analytical Research Division, J.R. DeVoe, Chief.

The statistical analysis of the certification data was performed by R.C. Paule of the NBS National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Table 1
Certified Concentrations of Constituent Elements in SRM's 2689, 2690, and 2691

----- Weight Percent-----			
Constituent Element	2689	2690	2691
Aluminum	12.94 ± 0.21	12.35 ± 0.28	9.81 ± 0.39
Barium	(0.08)	(0.65)	(0.66)
Calcium	2.18 ± 0.06	5.71 ± 0.13	18.45 ± 0.32
Iron (Total)	9.32 ± 0.06	3.57 ± 0.06	4.42 ± 0.03
Potassium	2.20 ± 0.03	1.04 ± 0.04	0.34 ± 0.01
Magnesium	0.61 ± 0.05	1.53 ± 0.05*	3.12 ± 0.08
Manganese	(0.03)	(0.03)	(0.02)
Sodium	0.25 ± 0.03	0.24 ± 0.02	1.09 ± 0.05
Phosphorus	0.10 ± 0.01	0.52 ± 0.01	0.51 ± 0.02
Silicon	24.06 ± 0.08	25.85 ± 0.17	16.83 ± 0.12
Sulfur	-----	0.15 ± 0.01	0.83 ± 0.05
Strontium	(0.07)	(0.20)	(0.27)
Titanium	0.75 ± 0.01	0.52 ± 0.01	0.90 ± 0.02
LOI 750 °C **	(1.76)	(0.53)	(0.23)
Moisture (110 °C) **	(0.14)	(0.12)	(0.08)
Residue on a 45 µm electroformed sieve (ASTM Standard Test Method C430-83)	12.8 ± 1.2	8.0 ± 0.7	10.5 ± 0.5

The uncertainty is expressed as two standard deviations of the certified value unless otherwise noted. *This uncertainty is based on scientific judgement. It is means to approximate ± two standard deviations of the certified value.

**In accordance with ASTM Standard Test Method C311-85.

Table 2
Multipliers For Element to Oxide Conversion.

Constituent Element	Oxide Sought	Multiplier
Aluminum	Al ₂ O ₃	1.88946
Barium	BaO	1.11650
Calcium	CaO	1.39919
Iron	Fe ₂ O ₃	1.42974
Potassium	K ₂ O	1.20459
Magnesium	MgO	1.65807
Manganese	MnO	1.29122
Manganese	Mn ₂ O ₃	1.43684
Sodium	Na ₂ O	1.34798
Phosphorus	P ₂ O ₅	2.29137
Silicon	SiO ₂	2.13931
Strontium	SrO	1.18261
Sulfur	SO ₃	2.49714
Titanium	TiO ₂	1.66806

Source and Preparation of Materials: The fly ashes were obtained from three different coal-fired power plants and are products of western Kentucky, Colorado, and Wyoming coals. Each fly ash was size classified and particles greater than 45 micrometers were removed. These coarse particles, mostly quartz and partially burned fragments, were ground to pass a No. 100 (150 μ m) sieve. This material was blended back into the rest of the fly ash, and the entire lot of material was homogenized in a ribbon blender, hermetically sealed in glass vials, and packaged. The packaging operations were performed in a temperature and humidity controlled atmosphere to minimize moisture differences between samples.

Homogeneity Testing: Stratified random selections of vials from each SRM were made and analyzed using x-ray fluorescence. Each vial was opened and two aliquots of 0.5 g were taken and fused into glass discs for XRF analyses. The duplicates of the individual vials made a total of 50 discs for each fly ash. For the elements measured (Al, Ca, Fe, and Si), no evidence of sample heterogeneity was observed.

TABLE 3
SUPPLEMENTAL INFORMATION ON THE SOURCE AND DESCRIPTION OF SRM's 2689, 2690, and 2691

	<u>2689</u>	<u>2690</u>	<u>2691</u>
Source:	Monier Resources, Inc. San Antonio, Texas 78216	Pozzolanic International Mercer Island, Wash.	Kansas City Power & Light Co. 98040 Kansas City, Mo. 64141
Power Plant:	Georgia Power Co. Plant Bowen Stilesboro, Ga.	Colorado-Ute Electric Association Craig Station Craig, Colo.	KCP and L Iatan Power Station Iatan, Mo.
ASTM C618 Class:	F	F	C
Coal Mine:	Western Kentucky Coal Districts 8 & 9	Trapper Mining, Inc. Craig, Colo.	Arco Black Thunder Mine Powder River Basin Gillette, Wyo.
Coal Type:	Bituminous (Moderate Sulfur)	Sub-bituminous (Low Sulfur)	Sub-bituminous (Low Sulfur)

Typical Properties of Coals Burned to Produce SRM's

Btu/lb:	12,000	9,700	8,800
Moisture, wt. %:	6	16.5	27.6
Ash, wt. %:	12	5.3	4.8
Sulfur, wt. %	1.5	0.3	0.3

TABLE 4
Analytical Techniques and Methods Used in the Certification of
SRM's 2689, 2690, and 2691

Method/ Element	A	B	C	D	E	F	G
Aluminum	*			*		*	
Barium	*			*			
Calcium	*			*	*	*	
Iron	*				*	*	
Magnesium	*					*	
Manganese		*		*		*	
Phosphorus		*			*	*	
Potassium	*			*		*	
Silicon	*	*	*			*	
Sodium	*			*		*	
Strontium	*			*		*	
Sulfur			*		*	*	
Titanium	*			*		*	
Loss on Ignition			*				
Moisture			*				
Sieve Residue (No.325 sieve)							*

ANALYTICAL TECHNIQUES AND METHODS

- A. Atomic Absorption
- B. Direct Current Plasma Emission Spectrometry
- C. Gravimetry
- D. Neutron Activation Analysis
- E. Titrimetry (Colorimetry)
- F. X-Ray Fluorescence Spectrometry
- G. ASTM Test Method C430-83 for Fineness of Hydraulic Cement by the 45-micrometer (No.325). Sieve as specified in ASTM C311, Standard Methods of Sampling and Testing Fly Ash or Natural Pozzolans for Use as an Admixture in Portland Cement Concrete.

Analysts—NBS Center for Analytical Chemistry

- 1. E. S. Beary
- 2. D. A. Becker
- 3. M. S. Epstein

Analysts—Construction Technology Laboratories, Portland Cement Association, Skokie, Illinois

- 1. Robert Crow
- 2. Howard M. Kanare
- 3. Hugh Love
- 4. Alyssa H. Malen
- 5. Charles M. Wilk

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1645

River Sediment

This Standard Reference Material is intended for use in the calibration of methods used in the analysis of river sediment and materials with similar matrices. The material has been freeze dried and is now essentially free from moisture. The certified values given below are based on measurements made on a dried sample of at least 100 mg for the trace elements and for a 1-g sample for iron and chromium.

The values are based on the results of 6 to 30 determinations by the analytical techniques indicated. The estimated uncertainties include those due to sample variation, possible method differences, and errors of measurement (see Preparation and Analysis).

Element	$\mu\text{g/g}$	Element	$\mu\text{g/g}$
Cadmium ^{c d}	10.2 ± 1.5	Thorium ^b	1.62 ± 0.22
Copper ^{b c}	109 ± 19	Uranium ^b	$1.11 \pm .05$
Lead ^{b d}	714 ± 28	Vanadium ^{a c}	23.5 ± 6.9
Manganese ^{b c}	785 ± 97	Zinc ^{a d}	1720 ± 169
Mercury ^{a c}	1.1 ± 0.5	<u>Weight %</u>	
Nickel ^{b d}	45.8 ± 2.9	Chromium ^{b c}	2.96 ± 0.28
Thallium ^b	1.44 ± 0.07	Iron ^{a c}	11.3 ± 1.2

^a Atomic Absorption Spectrometry

^b Isotope Dilution Mass Spectrometry

^c Neutron Activation Analysis

^d Polarography

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234
November 16, 1978

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Instructions for Use

The material, as received, is essentially free from moisture. In case of exposure to moisture, it should be dried without heat to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below -50°C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying in a desiccator over P_2O_5 or $\text{Mg}(\text{ClO}_4)_2$. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place.

Material of this kind is intrinsically heterogeneous. Consequently, the analyst should endeavor to minimize any segregation by thoroughly mixing the contents of the bottle by shaking and rolling before each use. In addition, when taking a portion for analysis, the analyst should strive to remove as representative a sample as possible.

Preparation and Analysis

This SRM was prepared from material dredged from the bottom of the Indiana Harbor Canal near Gary, Indiana. This material was screened to remove foreign objects, freeze dried, and sieved to pass a No. 80 (180 μm) screen. This material was thoroughly mixed in a V-blender, bottled, and sequentially numbered. The material has been radiation-sterilized to minimize alteration from biological activity.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles, some of them measuring replicate samples from each bottle. No correlation was found between measured values and the bottling sequence. The results of measurements on samples from different bottles did not appear to differ significantly from sub-samples within the bottles. Accordingly, it is believed that all bottles of this SRM have substantially the same composition. The analytical methods employed were those in regular use at NBS for certification of Standard Reference Materials, except as noted below. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative. The uncertainties of the certified values listed in the table include those associated with both measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

The following values have not been certified because either they are not based on results of a reference method, or were not determined by two or more independent methods. They are included for information only.

All values are in units of $\mu\text{g/g}$ of sample, unless otherwise indicated.

Antimony	(51)	Potassium	(1.2 wt. %)
Arsenic	(66)	Scandium	(2)
Cobalt	(8)	Sodium	(0.55 wt. %)
Lanthanum	(9)		

The values listed below are based on measurements made in one laboratory, and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the mean.

Kjeldahl Nitrogen	(0.0797% \pm 0.0048)
Total Phosphorus	(.051% \pm .0014)
Loss on Ignition (800 °C)	(10.72% \pm .28)
Oil and Grease (Freon)	(1.71% \pm .26)
Chemical Oxygen Demand (Dichromate)	(149,400 mg/kg \pm 9,000)

The methods used are:

Total Phosphorus - ASTM Method E-350.

Chemical Oxygen Demand (Dichromate) - Standard Methods for the Examination of Water and Waste Water, 14th Edition (1975), Section 508, page 550.

Oil and Grease (Freon 113 Extraction) - ibid., Section 502, page 518.

The following values are not certified, but are given to describe the matrix of the material: SiO₂ - 51%; MgO - 4%; Al₂O₃ - 4%; CaO - 4%.

H. L. Rook supervised collection, freeze drying, and homogenization of the SRM. The following members of the staff of the NBS Center for Analytical Chemistry performed the certification measurements: T. J. Brady; E. R. Deardoff; L. P. Dunstan; M. S. Epstein; R. Filby; M. Gallorini; E. L. Garner; T. E. Gills; J. W. Gramlich; R. R. Greenberg; S. H. Harrison; G. J. Lutz; L. A. Machlan; E. J. Maienthal; T. C. Rains; H. L. Rook; T. A. Rush; and W. P. Schmidt.

The development work, preceding the certification of the SRM, was supported by the Environmental Protection Agency under an Interagency Agreement.